

Contract No.: W912DQ-15-D-3013
Task Order No.: 002

U. S. Army Corps of Engineers
Kansas City District

**Final Bench Scale
Treatability Study Report,
Revision 1**

LCP Chemicals, Inc. Superfund Site
Remedial Design
Linden, Union County, New Jersey

July 18, 2018

**CDM
Smith**



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PROJECT: Contract No.: W912DQ-15-D-3013
Task Order No. 002
LCP Chemicals, Inc. Superfund Site
Remedial Design
Linden, New Jersey

SUBJECT: Final Bench Scale Treatability Study Report, Revision 1

Dear Travis:

CDM Federal Programs Corporation (CDM Smith) is pleased to submit the Final Bench Scale Treatability Study Report, Revision 1 for the LCP Chemicals Inc. Superfund Site, located in Linden, New Jersey. As requested, we are providing copies of this submittal as listed below. The report will also be posted to the project eRoom.

- Travis Young, USACE-KC District – 1 hard copy and 1 CD
- Jon Gorin, USEPA Region 2 – 1 CD

If you have any questions or comments concerning this submittal, please feel free to call me at (732) 590-4638.

Sincerely,

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Project Manager
CDM Federal Programs Corporation

cc: A. Rahmani, CDM Smith
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List of Acronyms

ASTM	ASTM International
BAL	Brooks Applied Labs
BR	Brooks Rand
bgs	below ground surface
CDM Smith	CDM Federal Programs Corporation
CLP	Contract Laboratory Program
cm/sec	centimeter per second
DPT	direct push technology
DTL	CDM Smith Denver Treatability Laboratory
g/cm ³	grams per cubic centimeter
g/mL	grams per milliliter
Hg	mercury
HgS	mercuric sulfide/mercury sulfide
LCP	LCP Chemicals Inc. Superfund Site
KC	Kansas City
kg/cm ²	kilograms per cubic centimeter
Ksp	solubility product constant
MBS	Molecular Bonding System
mg	milligrams
mg/kg	milligrams per kilogram
mm	millimeters
NIST	National Institute of Standard and Technology
ORP	oxidation reduction potential
PDI	pre-design investigation
psi	pounds per square inch
PTW	principal threat waste
RCRA	Resource Conservation and Recovery Act
RD	remedial design
ROD	Record of Decision
rpm	rotations per minute
RPD	relative percent difference
RSD	relative standard deviation
Site	LCP Chemicals Inc. Superfund Site
SDL	semi-dynamic leaching
SOP	standard operating procedure
SPLP	Synthetic Precipitation Leaching Procedure
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
µg/L	micrograms per liter
µg/m ³	micrograms per cubic meter
ZAAS	Zeeman atomic adsorption spectrometer

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Section 1

Introduction

Under the United States Army Corps of Engineers (USACE), Kansas City District (KC), Contract No. W912DQ-15-D-3013, Task Order No. 002, CDM Federal Programs Corporation (CDM Smith) has been tasked to support the USACE-KC and the United States Environmental Protection Agency (USEPA) in providing technical services, completing the pre-design investigation (PDI), bench scale investigation, pilot study, and remedial design (RD) at the LCP Chemicals Inc. Superfund Site (the Site) located in Linden, Union County, New Jersey. The PDI and PDI report (CDM Smith 2017a) have been completed. This document presents the Bench Scale Treatability Study.

1.1 Site Description

The Site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. In 1955, the General Aniline and Film Corporation constructed and began operating a chlor-alkali plant on the 26-acre property of the Site. Areas within the LCP site were leased to other companies for the operation of related manufacturing operations. In 1957, a western portion of the property was leased to Union Carbide Corporation to house a hydrogen plant operation that used byproducts of the chlorine production. That facility, known as the Linde Division hydrogen plant, operated until 1990. In addition, Kuehne Chemicals, Inc. leased an area on the northern portion of the property to manufacture sodium hypochlorite.

The chlor-alkali manufacturing operations ceased by 1985. The Hanlin Group, Inc. filed a petition under Chapter 11 of the bankruptcy code and liquidated its assets by 1994. As part of the bankruptcy, the Hanlin Group abandoned the LCP property; ownership reverted to the bankruptcy estate.

The Site was placed on the National Priorities List in 1998. In 1999, a potentially responsible party, ISP Environmental Services Inc., and USEPA entered into an Administrative Order to perform a Remedial Investigation and Feasibility Study. A Record of Decision (ROD) (USEPA 2014) was filed for the Site in February 2014.

1.2 Summary of the Record of Decision

The ROD for the Site (USEPA 2014) selected Alternative 4b to address contamination in groundwater, soil, sediments, and building material. Alternative 4b specifies the use of elemental sulfur in three different applications:

- **Capped Area:** The proposed cap consists of the following layers from top to bottom – soil layer (24 inches), impermeable geosynthetic membrane, and elemental sulfur layer (3 inches).
- **Building Debris:** Elemental sulfur is proposed to treat porous material with visible signs of elemental mercury; the treated porous material would be reduced in size and placed under the cap.

- **Principal Treat Waste (PTW):** Elemental sulfur is proposed to treat (stabilize) contaminated soil with visible elemental mercury by converting the elemental mercury to mercuric sulfide; specifically, the method of sulfur application is by in situ techniques (i.e., in situ stabilization). The final feasibility study (Cornerstone 2013) assumed only use of elemental sulfur with a range of 5 to 50 percent sulfur.

The ROD also states the following concerning treatment of the PTW:

- Stabilization would be accomplished by in situ mixing of elemental sulfur with PTW soil through the use of specialized mixing equipment (e.g., augers).
- The amount of elemental sulfur per volume of soil will be determined during the pre-design studies.
- The measure of success for the full-scale stabilization remedy would be the effectiveness of converting the elemental mercury to mercuric sulfide.
- The primary goal of stabilization would be to convert the elemental mercury to mercuric sulfide.

1.3 Bench Scale Treatability Study Objectives

As stated in the ROD, bench scale treatability study was conducted to determine the amount of elemental sulfur needed to convert elemental mercury to mercuric sulfide. This bench scale treatability study was expanded in scope based on a review of the effectiveness of elemental sulfur and other additives. The findings of the review are detailed in the memorandum titled “Use of Elemental Sulfur at the LCP Chemicals Inc. Superfund Site” dated August 19, 2016 (CDM Smith 2016). The review found that the mixing proposed in the ROD did not appear to be adequate to achieve the necessary mechanical energy and elevated temperature to impart enough energy to convert the elemental mercury to mercuric sulfide in the in-situ approach. Therefore, elemental sulfur alone using the proposed application processes (in situ mixing with augers) will most likely not be effective in converting elemental mercury in the PTW-contaminated soils to mercuric sulfide. To enable in situ conversion of elemental mercury to mercuric sulfide, use of reactive sulfides appear to be the most effective treatment additive.

The following additives and application methods were tested to determine their effectiveness in converting elemental mercury to mercuric sulfide:

- Elemental sulfur alone plus in situ auger mixing. This is the “baseline” additive and application specified in the ROD.
- Use of elemental sulfur in a rotary ball mill. This could be potentially used ex situ.
- Use of reactive sulfide for in situ applications. An evaluation of the effectiveness and a cost comparison was performed to select two reactive sulfides. The additives selected for testing were calcium polysulfide and FerroBlack® (Section 3.1).

In addition, solidification was performed in conjunction with stabilization to evaluate potential physical characteristics of the treated soil and the potential advantage of such characteristics

during full-scale implementation. Solidification is completed by adding cement for solidification and increase in strength, and bentonite is added for hydraulic conductivity reduction and facilitation of mixing during the auger in situ mixing process.

The bench scale treatability study was conducted following the steps and procedures detailed in the Final Bench Scale Treatability Study Work Plan (CDM Smith 2017b) and the associated standard operating procedures (SOP) including:

- SOP 1-1 Soil Sample Preparation
- SOP 1-2 Soil Mixing with Additives
- SOP 1-3 Elemental Mercury Analysis in Solid Samples
- SOP 1-4 Synthetic Precipitation Leaching Procedure and Semi-Dynamic Leaching Procedure on Stabilized Soils
- SOP 1-5 Unconfined Compressive Strength (Pocket Penetrometer)

Field and analytical activities in support of the bench scale treatability study were performed in accordance with the approved Quality Assurance Project Plan (CDM Smith 2017c).

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Section 2

Soil Sample Preparation and Baseline Analyses

2.1 Sample Collection and Compositing

Twenty boreholes were advanced from 0 to 19 feet below ground surface (ft bgs) using direct push technology (DPT) at the bench scale study area (Figure 2-1) on April 11, 2017 through April 14, 2017 to locate soil where visible elemental mercury was identified during the remedial investigation.

At each of the twenty DPT borings, 5-foot-long, 2-inch outer diameter macro-cores were collected from undisturbed soil in advance of the DPT drill rods. Upon retrieval, each core was visually inspected by the geologist for the presence of visible elemental mercury, and the soils were screened for mercury vapors using a Jerome® 431-X mercury vapor analyzer.

Mercury vapor readings at the twenty boring locations ranged from 0.0 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) to above the maximum range of the Jerome® 431-X mercury vapor analyzer (0.999 $\mu\text{g}/\text{m}^3$). No visible elemental mercury was observed in any of the soil borings.

Two soil samples were collected in 5-gallon buckets from the pilot study area. The initial plan was to collect 5-gallons of soil for a high mercury sample (designated as SS-H) that contained visible elemental mercury, and to collect a medium mercury sample (designated as SS-M) with soil that had mercury vapor readings in the range of 0.5 $\mu\text{g}/\text{m}^3$ to 0.999 $\mu\text{g}/\text{m}^3$. However, because no visible elemental mercury was observed, the decision was made to have each sample represent a complete 0-19 ft bgs soil column, and then to artificially spike each sample with mercury in the CDM Smith Denver Treatability Laboratory (DTL) in accordance with SOP 1-1, Soil Sample Preparation. Each of the two 5-gallon buckets of soil represented one complete borehole run from 0 to 19 ft bgs. However, in addition to soil from a complete borehole, the medium sample contained approximately 1-gallon of soil from another borehole with mercury vapor readings greater than 0.999 $\mu\text{g}/\text{m}^3$. Samples were sent to the DTL. All twenty DTP boreholes were abandoned by backfilling with cement-bentonite.

Upon receipt at the DTL, the soil was transferred from the 5-gallon buckets into stainless steel mixing bowls. The two composite soil samples (SS-M and SS-H) were prepared using SOP 1-1, Soil Sample Preparation. Briefly, the soil was air dried in a fume hood for 14 days. The dried soil was disaggregated using a mortar and pestle and passed through a #10 US Sieve No. plastic sieve. Each soil sample was homogenized and split into 250-gram subsamples using a riffle splitter.

2.2 Sample Spiking

Subsamples of the composited samples were obtained and analyzed for elemental mercury at the DTL according to SOP 1-3, Elemental Mercury Analyses in Solid Samples. Measured results for SS-M by this method were 115 milligrams per kilogram (mg/kg) elemental mercury. For soil sample SS-H elemental mercury was measured at 275 mg/kg.

In accordance with the Bench Scale Treatability Study Work Plan the target mercury concentrations for the composite samples were 1,500 mg/kg for SS-M and 6,000 mg/kg for SS-H. Composite samples did not meet the target elemental mercury concentrations, and therefore, elemental mercury levels were augmented to achieve the target starting concentrations. As described in SOP 1-1, Section 5.3, Mercury Addition, the entire composited soil sample was to be spiked to the required elemental mercury concentration. SOP 1-1 was modified to spike a subsample of the soil prior to each treatability study test; i.e., 250 g aliquots of SS-M and SS-H were spiked with 375 milligrams (mg) (= 1,500 mg/kg = SS-M) and 1500 mg (= 6,000 mg/kg = SS-H) of elemental mercury, respectively. The modification to the SOP was reviewed and approved. All the spiked samples were placed on a rotary tumble that rotated the bottles at approximately 30 rotations per minute (rpm) for between 18 and 24-hours to equally distribute the elemental mercury.

2.3 Mercury Analysis Method Development on Composite Soil

The following discussion outlines the process used to determine different forms or species of mercury at the DTL using an Ohio Lumex PYRO-915+ coupled to a RA-915M, following SOP 1-3, Elemental Mercury Analysis in Solid Samples. This instrument uses varying temperatures to volatilize mercury in a solid sample and quantify it using an inline spectrometer. The PYRO-915+ heats the sample to 150-210°C to volatilize the elemental mercury, and the mercury vapor is transported to the RA-915M where it is analyzed by a Zeeman atomic adsorption spectrometer (ZAAS). The boost heating mode is then employed to heat the sample to 450-600°C to volatilize the remaining forms of mercury present. This method was developed on composite samples SS-M and SS-H.

2.3.1 Mode Selection and Standard Sample Testing

In accordance with SOP 1-3 Mercury Analysis in Solid Samples, Mode 8 (temperature 150 to 210°C) was used to quantify elemental mercury, and Mode 2 (temperature 520 to 580°C) was used to quantify black metacinnabar (beta-mercuric sulfide [HgS]) and red cinnabar (alpha-HgS). In practice, the instrument was operated in Mode 8, and a manual temperature boost was initiated to raise the temperature of the test cell to 450 to 600°C. Calibration of the instrument was accomplished through the analysis of National Institute of Standard and Technology (NIST) soil standard 2711 (total mercury of 32.6 mg/kg), sand spiked with elemental mercury and black and red cinnabar reagent grade HgS. The elemental mercury and HgS spiked materials were performed to demonstrate adequate separation of the forms of mercury to be measured. The HgS (red and black cinnabar) was purchased from Fischer Scientific and packaged through Alfa Aesar with a purity 99.999 percent.

Figure 2-2 provides the spectral graph of elemental mercury in sand at 1,000 mg/kg. The spiked sand is inserted into the combustion chamber at 50 seconds, and the temperature boost was initiated at 300 seconds. As can be observed in the figure, elemental mercury vaporizes immediately, at approximately 60 seconds.



Figure 2-2 Elemental Mercury in Sand

Figure 2-3 provides the spectral graph of black metacinnabar (beta-HgS) in sand at 1,000 mg/kg. The sample was inserted at 50 seconds, and the temperature boost was initiated at 300 seconds. As shown in the figure, the instrument response for beta-HgS is at an approximate time of 450 seconds (i.e., the HgS volatilizes producing elemental mercury).

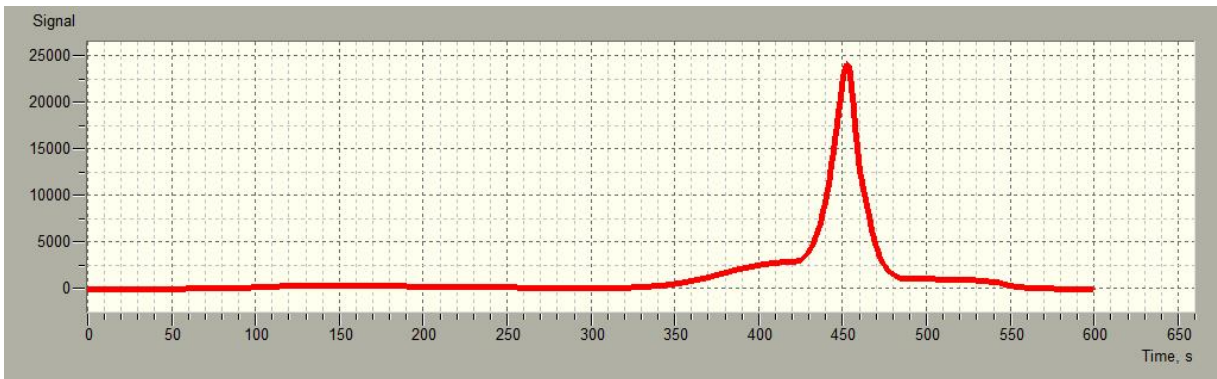


Figure 2-3 Black Cinnabar (beta-HgS) in Sand

Figure 2-4 provides the spectral graph of red cinnabar (alpha-HgS) in sand at 1,000 mg/kg. The sample was inserted into the combustion chamber at 50 seconds, and the temperature boost was initiated at 300 seconds. As shown in the figure, alpha-HgS volatilizes at approximately 450 seconds.

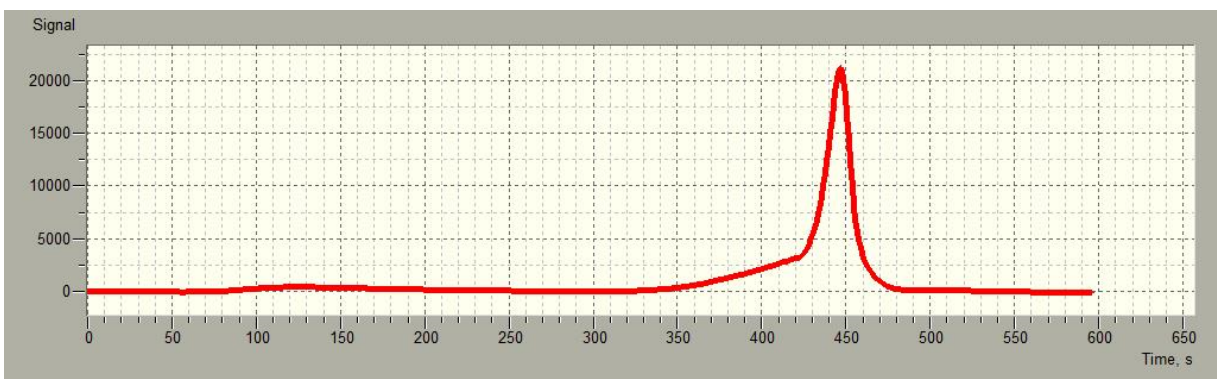


Figure 2-4 Red Cinnabar (alpha-HgS) in Sand

Figure 2-5 (below) provides the spectral graph of red cinnabar (alpha-HgS) and black metacinnabar (beta-HgS) in sand each at 1,000 mg/kg. The sample was inserted into the combustion chamber at 50 seconds, and the temperature boost was initiated at 300 seconds. As shown in the figure, both alpha-HgS and beta-HgS volatilize at approximately 450 seconds.

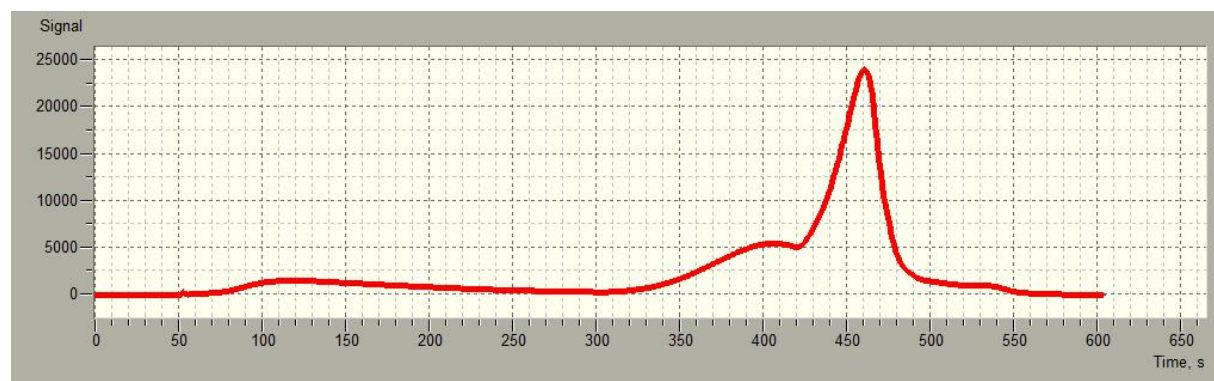


Figure 2-5 Red Cinnabar (alpha-HgS) and Black Cinnabar (beta-HgS) in Sand

These results demonstrate that the instrument can successfully differentiate between elemental mercury and forms of mercury sulfide (cinnabar). However, adequate separation between black and red cinnabar cannot be observed.

Individual subsamples (i.e., standards) of elemental mercury, black cinnabar, and red cinnabar were created at concentrations of 1,000 mg/kg in sand. Five individual analyses were performed to assess homogeneity of the subsamples after spiking and mixing. The average concentration, the percent recovery and the percent relative standard deviation (RSD) of the 5 analyses are summarized in the table below.

Standard	Average Concentration (mg/kg)	Recovery (percent [%])	RSD (%)
alpha-HgS (Red) in Sand	978	97.8	17
beta-HgS (Black) in Sand	950	95	9
Elemental mercury (Hg) in Sand	315	31.5	83

The average results for the mercury sulfides (alpha-HgS and beta-HgS) were acceptable with recoveries above 95 percent and RSDs were 17 and 9 percent for the red and black cinnabar, respectively. These forms of mercury were mixed with the sand as pure dry powders and evenly distributed. Elemental mercury average recoveries were 31 percent with an RSD of 83 percent. The elemental mercury may form small droplets that are difficult to evenly distribute in sand, creating heterogeneity issues (high RSD value) and low recovery (low average concentration). This effect was not observed in soils and treated samples to the extent observed in sand.

Based on these results, the method is able to provide reproducible results for total mercury; however, more variability is present in the quantitation of mercury sulfides. The soils did not exhibit significant differences in total mercury content.

2.4 Baseline Chemical Characterization

Subsamples of each un-spiked and spiked final composite sample (SS-M and SS-H) were analyzed for a variety of baseline characteristics. Sample analyses were completed via a combination of Tier 2 (Contract Laboratory Program [CLP]) and Tier 4 (DTL, and CDM Smith-subcontracted laboratories). Results from the baseline characterization of the soil samples are summarized below.

2.4.1 Mercury Analysis of Un-Spiked Soil Samples

Both un-spiked prepared soils were analyzed following the method developed to determine a baseline concentration for elemental and mercury sulfides (SOP 1-3 modified as discussed in Section 2.3.1). The results for the samples are presented in two different ways: 1) total mercury - which quantifies all forms of mercury (the combination of pre- and post-temperature boost or elemental mercury plus mercury sulfides), and 2) mercury sulfides - which quantifies black and red cinnabar (and possibly other types of more stable mercury species) from the post boost application. The table below provides these results as average concentrations and the RSD for 5 separate analyses performed.

Sample	Total Mercury		Mercury Sulfides	
	Average Concentration (mg/kg)	RSD (%)	Average Concentration (mg/kg)	RSD (%)
SS-M	502 (115 elemental Hg + 387 mercury sulfides)	19	387	41
SS-H	514 (275 elemental Hg + 239 mercury sulfides)	21	239	28

2.4.2 Mercury Analysis of Spiked Soil Samples

Analysis of each spiked composite samples before addition of stabilization amendments was completed per the modifications to SOP 3-1 as described in Section 2.3.1. The table below provides results of the spiked samples (SS-H and SS-M) performed by the DTL.

Sample	Elemental Mercury		Mercury Sulfides	
	Average Concentration (mg/kg)	RSD (%)	Average Concentration (mg/kg)	RSD (%)
SS-H-6000	4251	3	356	28
SS-M-1500	1143	9	315	24

These samples were analyzed by the Ohio Lumex mercury analyzer and the results are provided for elemental mercury and mercury sulfide fractions (total Hg = the sum of these two fractions). Because a small aliquot of soil is used during the analysis (5 – 20 mg), five individual analyses were performed to assess homogeneity of the soil after spiking and mixing. The results reported in the table above are an average of the 5 analyses and the % RSD of the five results is also provided. As shown, total mercury was 4,607 mg/kg (4,251 + 356) for SS-H-6000 and 1,458 (1,143 + 315) for SS-M-1500. This compares to 6,990/6,910 mg/kg and 2,300 mg/kg for SS-H-6000 and SS-M-1500, respectively, from the subcontract laboratory (see Table 2-1 and discussion

in next section). Given the difficulty in creating uniform subsamples with spiked elemental mercury, these values are acceptable and clearly show the differences between the SS-M-1500 and SS-H-6000 concentrations and the differences between elemental mercury and mercury sulfides. In addition, the percent RSDs were excellent for elemental mercury (9 percent and 3 percent) and good for HgS (24 percent and 28 percent).

2.4.2 Chemical Characterization

Chemical characterization was performed on both the un-spiked and spiked composite samples before treatment using the following analyses:

- Target Analyte List (TAL) metals: Digestion by SW-846 3050A, analyses by SW-846 6020B/7471B – CLP (Tier 2).

Additional chemical characterization was performed on the spiked composite samples before treatment using the following analyses:

- Mercury Speciation (to identify forms of mercury present): 6 fractions according to SOP Brooks Rand (BR)-0013 (or equivalent) – CDM Smith subcontract laboratory (Tier 4).
- Leachability: A modified synthetic precipitation leaching procedure (SPLP) was conducted at the DTL (see SOP 1-4, SPLP and Semi-Dynamic Leaching Procedure on Stabilized Soils). The generated leachate was analyzed for the 8 Resource Conservation and Recovery Act (RCRA) metals – CLP (Tier 2).

Table 2-1 and Table 2-2 provide the results for the TAL metals and mercury speciation, respectively (SPLP results are discussed in Section 7). As previously discussed, sample SS-H was spiked with 6,000 mg/kg elemental mercury and sample SS-M was spiked with 1,500 mg/kg elemental mercury. These samples were analyzed for TAL metals and mercury speciation as discussed above and according to Section 2.3 of the approved Bench Scale Treatability Study Work Plan. Five extracts (F1-F5) for mercury speciation were performed in addition to volatile elemental mercury (F0): F1, Water soluble = HgCl_2 , (HgO and HgSO_4); F2, Weak-acid soluble = HgO (HgSO_4); F3, Organo-complexed = Hg-humic acid complexes, CH_3Hg , Hg_2Cl_2 ; F4, Strongly complexed and elemental = Elemental Hg, Hg_2Cl_2 ; F5, Mineral-bound = HgS , HgSe , HgAu . Species in “(---)” indicate minor species reporting to the fraction or reporting to multiple fractions.

As shown on Table 2-1, total mercury was 2,300 mg/kg (J qualified) in SS-M-1500 and 6,990 mg/kg (J qualified) in SS-H-6000 (duplicate for SS-H-6000 was 6,910 mg/kg [J qualified]). As shown in table included in Section 2.4.1, the baseline (un-spiked) concentrations measured at the DTL using SOP 1-3 in SS-M-1500 was 502 mg/kg total mercury and the baseline in SS-H-6000 was 514 mg/kg total mercury. For SS-M-1500 the total mercury concentration should be 2,002 mg/kg ($1500 + 502 = 2,002$ mg/kg) and for SS-H-6000 the concentration should be 6,504 mg/kg ($6,000 + 514 = 6,514$ mg/kg). Given the difficulty in homogenizing and uniformly distributing elemental mercury in soils, the total mercury values are in good agreement between the sum of the baseline (un-spiked) and spiked concentrations (2,002 and 6,514 mg/kg) compared to the subcontract laboratory values (2,300 and 6,990/6,910 mg/kg).

As shown in Table 2-2, the elemental mercury should have been extracted into fraction F4 during the speciation analyses. However, the speciation analyses showed only 26.6 percent (SS-M-1500) and 46.8/48.8 percent (SS-H-6000) of the mercury reporting to fraction F4. These values are low and reflect the “J” qualification for the speciation analysis. In addition, the sum of the fractions for SS-M-1500 is much higher (4,066 mg/kg) compared to the total mercury (2,300 mg/kg). See Section 7 for additional discussion concerning the mercury speciation results.

The SPLP data are reported and discussed in Section 6.

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Section 3

Addition of Stabilization Additives

3.1 Reactive Sulfide Comparison and Selection

As previously summarized in the memorandum titled “Use of Elemental Sulfur at the LCP Chemicals Inc. Superfund Site” dated August 19, 2016 (CDM Smith 2016), to enable in situ conversion of elemental mercury to mercuric sulfide, reactive sulfides appear to be the most effective treatment additive. In accordance with the Bench Scale Treatability Study Work Plan (CDM Smith 2017b), the 2 additives selected were calcium polysulfide and FerroBlack®. The additives were compared and selected based on the Additive Comparison Table, which is attached in Appendix A. Therefore, the following combinations of additives and application methods were tested:

- Elemental sulfur and in situ auger mixing
- Elemental sulfur and ex situ ball mill processing
- Reactive sulfide No. 1 and in situ auger mixing (calcium polysulfide)
- Reactive sulfide No. 2 and in situ auger mixing (FerroBlack®)

Details of the addition of the additives, concentrations of the additives, and processing methods are provided in SOP 1-2, Soil Mixing with Additives. Each of the combinations and applications are summarized in the following sections. Table 3-1 provides the sample identification descriptions as well as the soil used (SS-M or SS-H), the amendment and the targeted concentration of the amendments. In total, 29 mixtures were tested.

3.2 Elemental Sulfur and In Situ Auger Mixing

In accordance with the Bench Scale Treatability Study Work Plan (CDM Smith 2017b), for spiked soil SS-H-6000, sulfur was added at concentrations of 5 and 25 weight percent of the soil, and for spiked soil SS-M-1500, sulfur was added at concentrations of 5, 12.5, and 25 weight percent. The soil/sulfur mixtures were mixed in a standard paddle mixer at 10 to 15 rpm for 90 minutes. This process best represents in situ mixing with large diameter augers. Table 3-2 presents the soil and amendment masses prepared for this test.

3.3 Elemental Sulfur and Rotary Ball Mill Processing

Based on review of literature to create HgS using mechanical energy in an ex situ process, sulfur should be added to the composite soils in a 1:1 weight ratio of the concentration of elemental mercury in the soil (i.e., 1,500 mg/kg and 6,000 mg/kg of sulfur would be added to spiked samples SS-M-1500 and SS-H-6000, respectively) (Lopez 2008). This represents an excess sulfur content of 6.25 times the stoichiometric ratio based on the conversion of elemental mercury to HgS. To provide a range of sulfur addition, sulfur was added to SS-M at concentrations of 1,000, 1,500 and 2,000 mg/kg. Sulfur was added to SS-H at concentrations of 4,000, 6,000, and 8,000

mg/kg. To simulate potential full-scale ex situ mixing, the mixtures were placed in a rotary tumbler mill with agate balls (i.e., simulating a commercial rotary ball mill) and processed for 90 minutes at approximately 70 rpm. Table 3-3 presents the soil and amendment masses prepared for this test.

3.4 Reactive Sulfide No. 1 and In Situ Auger Mixing (Calcium Polysulfide)

Calcium polysulfide was selected as the first reactive sulfide to test, and was obtained from the commercial vendor Graus Chemicals. Calcium polysulfide is commercially available as a 29-weight percent solution. In accordance with the Bench Scale Treatability Study Work Plan (CDM Smith 2017b), calcium polysulfide was added to SS-M-1500 at concentrations of 1.5, 3, and 5 weight percent (weight percent based on mass of commercial product as purchased relative to the mass of soil; therefore, resulting concentrations were 0.435, 0.87, and 1.45 weight percent, respectively), and to SS-H-6000 at 2, 6, and 10 weight percent, in accordance with SOP 1-2. The mixtures were processed to simulate in situ mixing with large diameter augers (mixed in paddle mixer at 10 to 15 rpm for 90 minutes).

Incomplete mixing (distribution) and “clumping” of the calcium polysulfide was observed during the mixing process. This was probably due to the relatively high viscosity of the 29 percent calcium polysulfide. A more aggressive mixing procedure may have been successful at breaking up the clumps and distributing the amendment more evenly throughout the soil but would not have simulated the anticipated procedures that would be implemented during full scale remedial activities. Therefore, additional tests were performed where an equal volume of tap water was added to the calcium polysulfide prior to addition to the spiked soil to reduce the viscosity of the amendment and allow for a more homogeneous distribution throughout the soil. These tests were performed using the highest concentrations of calcium polysulfide for each soil (5 percent for SS-M and 10 percent for SS-H). The sample IDs for these tests have been appended with a “-OPTI” (OPTI = optimized mixing).

Table 3-4 presents the soil and amendment masses prepared for these tests.

3.5 Reactive Sulfide No. 2 and In Situ Auger Mixing (FerroBlack®)

FerroBlack® was selected as the second reactive sulfide to test. FerroBlack® is a commercial product consisting of microscale iron sulfide and was provided by REDOX Solutions (Carmel, Indiana). Based on the recommendations of REDOX Solutions, stabilization with FerroBlack® was tested with and without the addition of calcium oxide. Calcium oxide is used to raise the temperature of the soil (i.e., exothermic reaction with soil) and increase effectiveness of the FerroBlack®. For samples tested with calcium oxide, 10 weight percent (final mixture is 10 percent calcium oxide and 90 percent soil by weight) was added to each sample, and mixed using a paddle mixer. Once a temperature of 90 degrees Fahrenheit had been reached, FerroBlack® was added to SS-M-1500 and SS-H-6000 at a concentration of 5 weight percent (final mixture is 5 percent FerroBlack® and 95 percent soil by weight) in accordance with SOP 1-2. For samples tested without calcium oxide, FerroBlack® was added to SS-M-1500 at concentrations of 2, 5, and

10 weight percent, and to SS-H-6000 at 5, 10, and 15 weight percent. The mixtures were processed to simulate in situ mixing with large diameter augers (mixed in paddle mixer at 10 to 15 rpm for 90 minutes).

Incomplete mixing (distribution) and “clumping” of the FerroBlack® was observed during the mixing process (similar to the calcium polysulfide tests). This was probably due to the relatively high viscosity of the FerroBlack® and the small volume added when compared to the mass of soil treated. A more aggressive mixing procedure may have been successful at breaking up the clumps and distributing the amendment evenly throughout the soil but would not have simulated the anticipated procedures that would be implemented during full scale remedial activities.

Therefore, additional tests were conducted where an equal volume of tap water was added to the FerroBlack® prior to addition to the spiked soil to reduce the viscosity of the amendment and allow for a more homogeneous distribution throughout the soil. These tests were performed using the highest concentrations of FerroBlack® (10 percent for SS-M and 15 percent for SS-H) for each soil. The sample IDs for these tests have been appended with a “-OPTI” (OPTI = optimized mixing).

Table 3-5 presents the soil and amendment masses prepared for these tests.

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Section 4

Analyses of Elemental Mercury in the Stabilized Soil Mixtures

The main performance criterion for the bench scale study is the conversion of the elemental mercury to mercuric sulfide. Each of the 29 mixtures discussed in Section 3, Table 3-1 were analyzed for elemental mercury and mercury sulfide at the DTL using the procedure detailed in SOP 1-3, Analyses of Elemental Mercury in Solid Samples. The procedures in SOP 1-3 were modified from using both Mode 2 and Mode 8 for analysis, to utilizing Mode 8 with heating boost. The method development and deviations from SOP 1-3 are detailed in Section 2.3.1.

The concentration of elemental mercury in the stabilized samples was compared to the initial concentrations (as measured by SOP 1-3), and a percent decrease in elemental mercury calculated. This percent decrease was assumed to be the percent of conversion of the elemental mercury to mercuric sulfide (either or both red cinnabar or black cinnabar forms).

$$\text{Percent Conversion} = \frac{\text{Pretreat Elemental Hg} - \text{Posttreat Elemental Hg}}{\text{Pretreat Elemental Hg}} \times 100\%$$

For example, in Table 4-1 the percent conversion of elemental mercury to mercury sulfide based on a decrease of elemental mercury was calculated to be 35.9 percent in sample SS-F-S-5.

In addition, the amount of mercury sulfide in each stabilized sample was determined and compared to the initial elemental mercury and mercury sulfide concentrations. The amount of additional mercury sulfide measured in the stabilized samples (compared to initial amount) was determined. The amount of additional mercury sulfide divided by the initial elemental mercury concentration was calculated as a percent conversion to mercury sulfide.

$$\text{Percent Conversion} = \frac{\text{Posttreat HgS} - \text{Pretreat HgS}}{\text{Pretreat Elemental Hg}} \times 100\%$$

For example, in Table 4-1 the percent conversion of elemental mercury to mercury sulfide based on an increase of mercury sulfide was calculated to be 5.8 percent in sample SS-F-S-5.

The two values (percent decrease of elemental mercury and percent of mercury sulfide increase) were used as the primary performance criteria for evaluation of effectiveness of each treatment mixture.

4.1 Elemental Sulfur and In Situ Auger Mixing

As discussed in Section 3.2, elemental sulfur was added to spiked soil SS-M-1500 at three different concentrations (5 percent, 12.5 percent, and 25 percent) and to spiked soil SS-H-6000 at two concentrations (5 percent and 25 percent) and mixed with a paddle mixer to simulate in situ auger mixing. Both pre-treated and post-treated soils were analyzed at the DTL for elemental

mercury and mercury sulfides. As discussed above, a conversion to HgS based on elemental mercury decrease and a conversion to HgS based on an increase of mercury sulfides were calculated. These results are presented in Table 4-1. The last two columns in the table show the two conversion values

Treatment with the highest amount of elemental sulfur (25 percent) was the most successful at elemental mercury conversion for SS-H-6000 and was the most successful using 12.5 percent elemental sulfur for SS-M-1500. However, a large difference exists between the two conversion values. For example, the treatment of SS-M-1500 (spiked with 1500 mg/kg elemental mercury) with 12.5% elemental sulfur resulted in 73.2 percent conversion based on the increase of mercury sulfides, and a 22.9 percent conversion based on the decrease of elemental mercury. Treatment of SS-M-1500 and SS-H-6000 with 25 percent elemental sulfur resulted in 17.4 percent and 56.6 percent conversion (based on the decrease of elemental mercury) and 67.5 percent and 19 percent conversion (based on the increase of mercury sulfides), respectively.

4.2 Elemental Sulfur and Rotary Ball Mill Processing

Section 3.3 discusses the addition of elemental sulfur and processing with a rotary ball mill to simulate ex situ mixing. Elemental sulfur was added to spiked soil SS-M-1500 at three different concentrations (1,000 mg/kg, 1,500 mg/kg, and 2,000 mg/kg) and to spiked soil SS-H-6000 at three concentrations (4,000 mg/kg, 6,000 mg/kg, and 8,000 mg/kg). Both pre-treated and post-treated soils were analyzed at the DTL for elemental mercury and mercury sulfides. A conversion based on elemental mercury decreases and a conversion based on an increase of mercury sulfides was calculated. These results are presented in Table 4-2.

Based on the analytical results produced at the DTL, very little elemental mercury was converted to mercury sulfide. Based on measurements of mercury sulfides (last column in Table 4-2), between 2.2 percent and 8.3 percent of the elemental mercury was converted to HgS. Based on measurements of elemental mercury, the values ranged from 11.8 percent to 31.6 percent conversion.

4.3 Reactive Sulfide No. 1 and In Situ Auger Mixing (Calcium Polysulfide)

Section 3.4 discusses the addition of calcium polysulfide and mixing with a paddle mixer to simulate in situ auger mixing. Calcium polysulfide was added to spiked soil SS-M-1500 at three different concentrations (1.5 percent, 3 percent, and 5 percent) and to spiked soil SS-H-6000 at three concentrations (2 percent, 6 percent, and 10 percent). In addition, two of the mixtures, SS-M-1500 with 5 percent calcium polysulfide and SS-H-6000 with 10 percent calcium polysulfide, were repeated to address observed clumping and poor mixing during the tests. These tests were optimized by diluting calcium polysulfide in an equal volume of tap water prior to addition and mixing.

Both pre-treated and post-treated soils were analyzed at the DTL for elemental mercury and mercury sulfides. A conversion based on elemental mercury decreases and a conversion based on an increase of mercury sulfides was calculated. These results are presented in Table 4-3.

Based on the analytical results produced at the DTL, conversion of elemental mercury to mercury sulfide was typically more effective at higher calcium polysulfide concentrations for both SS-M-1500 and SS-H-6000. The best performing mixtures for each soil were the mixtures that had been optimized (“OPTI” samples) through the addition of tap water prior to addition and mixing. Based on measurement of mercury sulfides, the mixtures for SS-M-1500 with calcium polysulfide at 5 percent and an equal volume of water showed conversion to mercury sulfide of 93.6%. The mixture for SS-H-6000 with calcium polysulfide at 10 percent and an equal volume of tap water exhibited a conversion to mercury sulfide of 80.2 percent based on measurement of mercury sulfides. Based on measurements of elemental mercury, the same two optimized mixtures exhibited conversions of 84.4 percent and 93.4 percent, respectively for SS-M and SS-H. The two methods used to calculate the percent conversion to mercury sulfide are in good agreement for the optimized samples providing confidence in the overall conclusions concerning the effectiveness of stabilization.

4.4 Reactive Sulfide No. 2 and In Situ Auger Mixing (FerroBlack®)

Section 3.5 discusses the addition of FerroBlack® and mixing with paddle mixer to simulate in situ auger mixing. FerroBlack® was added to spiked soil SS-M-1500 at three different concentrations (2 percent, 5 percent, and 10 percent) and to spiked soil SS-H-6000 at three concentrations (5 percent, 10 percent, and 15 percent). Calcium oxide was added to two of the mixtures at 10 percent soil mass to increase the soil temperature above 90 degrees Fahrenheit per instructions from the manufacturer. In addition, two of the mixtures, SS-M-1500 with 5 percent FerroBlack® and SS-H-6000 with 10 percent FerroBlack® were repeated to address observed clumping and poor mixing during the tests. These tests were optimized by diluting FerroBlack® in an equal volume of tap water prior to addition and mixing.

Both pre-treated and post-treated soils were analyzed at the DTL for elemental mercury and mercury sulfides. A conversion based on elemental mercury decreases and a conversion based on an increase of mercury sulfides were calculated. These results are presented in Table 4-4.

Based on the measurements of mercury sulfides produced at the DTL (last column in Table 4-4), conversion of elemental mercury to mercury sulfide was most effective for SS-M-1500 when treated with optimized 5 percent FerroBlack® (94.6 percent) and most effective for SS-H-6000 when treated with 10 percent FerroBlack® (97.9 percent). When SS-H-1600 was treated with optimized 15 percent FerroBlack® (with tap water), the conversion of 95.6% was similar to the level observed in 10 percent FerroBlack® mixture. Based on measurements of elemental mercury, the percent conversions were very similar compared to the values based on measurements of mercury sulfide for the two optimized mixtures (92.9 percent compared to 94.6 percent for SS-M-1500 with 5 percent FerroBlack®, and 92.0 percent compared to 95.6 percent for SS-H-6000 15 percent FerroBlack®). The excellent agreement provides confidence in the overall conclusions concerning the effectiveness of stabilization. The use of calcium oxide improved the conversion based on elemental mercury; however, the results were typically similar to the optimized samples.

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Section 5

Solidification Tests

Based upon the evaluation of the conversion of elemental mercury to mercuric sulfide, 6 of the most effective mixtures (conversion to mercuric sulfide using in situ techniques) were selected for solidification tests (see SOP 1-2 for details). Table 5-1 provides this list of sample mixtures and the concentrations of solidification material (cement and bentonite) used. The sections below summarize the selection of the stabilization mixtures and the procedures used to solidify the mixtures. In addition, the method utilized for solidification, as well as non-leaching chemical analyses of the stabilized and solidified samples.

5.1 Comparison and Selection

Table 5-1 provides the list of stabilization mixtures selected for solidification. As discussed in Section 4, calcium polysulfide and FerroBlack® performed the best when compared to the elemental sulfur amendments in conversion of elemental mercury to mercury sulfide. The best performing concentrations of calcium polysulfide and FerroBlack® in each mixture were selected based on percent conversion to mercury sulfide. In addition, the two mixtures of FerroBlack® with additions of calcium oxide were selected based on increased conversion rates compared to the mixtures without the calcium oxide.

5.2 Mixing, Curing and Results

As shown in Table 5-1, composite soil samples (SS-M-1500 and SS-H-6000) were stabilized and solidified by adding the selected stabilization agent, water, between 5 percent and 10 percent cement, and between 2.5 percent and 5 percent bentonite (weight percentages based on mass of soil). The optimized stabilization procedure involved the addition of water to more effectively distribute the amendment, because solidification also involves the addition of water, the processes are similar (optimized). The stabilized and solidified mixtures were placed in 2- x 3-inch plastic cylinders and let cure for a minimum of 7 days. The unconfined compressive strength was measured using a pocket penetrometer (see SOP 1-5) at the DTL. All solidified samples exhibited an unconfined compressive strength of greater than 4.5 kilograms per square centimeter (kg/cm²) which is the maximum measured value for the pocket penetrometer.

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Section 6

Additional Testing of Stabilized and Solidified Mixtures

Additional testing of stabilized, and stabilized/solidified mixtures were performed in accordance with the approved Bench Scale Treatability Study Work Plan (CDM Smith 2017b). The stabilized mixtures (10 samples) were selected based on the best conversions of elemental mercury to mercury sulfide for sample amended with FerroBlack® (4 samples), calcium polysulfide (4 samples) and sulfur (2 samples). In addition, three of the stabilized and solidified samples were selected for testing identified below. Table 6-1 lists the mixtures selected for additional testing, and includes a description of the stabilization and solidification amendments as well as the additional tests that were performed. Although the work plan specified eight stabilized samples for testing, an additional 2 stabilized samples were selected for the testing. Table 6-1 also show the composite samples SS-H-6000 and SS-M-1500 that were analyzed for comparison to the treated mixtures. The following tests were conducted on all the 12 samples:

- TAL Metals – Analyzed by Chemtech Consulting Group in Mountainside, New Jersey, a contracted CLP laboratory.
- Leachability: A modified SPLP was conducted at the DTL (see SOP 1-4, SPLP, and Semi-Dynamic Leaching Procedure on Stabilized Soils). The generated leachate was analyzed for the 8 RCRA metals by CLP laboratory.
- Regulatory Classification including corrosivity (pH) and reactivity (hydrogen sulfide generation at pH 2) – Measurements conducted at the DTL.

In addition, three of the stabilized and solidified samples (during the solidification process, water was added similarly to the optimized stabilization process) were selected for testing geotechnical properties and semi-dynamic leaching based on percent mercury conversion during the stabilization testing (Section 5). The three samples, SS-H-6000 with 10 percent calcium polysulfide, SS-H-6000 with 15 percent FerroBlack®, and SS-H-6000 with 10 percent FerroBlack® and 10 percent calcium oxide were selected for the additional tests after a cure time of 28 days. The tests included:

- Leachability: A semi-dynamic leaching (SDL) test on the three stabilized and solidified materials identified above using a combination of SW-846 1315 and ASTM International (ASTM)-C1308 (9 leaching times, 2 hours to 42 days, see SOP 1-4) was conducted at the DTL. The leachate was analyzed for dissolved mercury by Chemtech Consulting Group.
- Unconfined compressive strength by ASTM C39M – CDM Smith Boston Geotechnical Laboratory
- Hydraulic conductivity by ASTM D5084 – CDM Smith Boston Geotechnical Laboratory

- Moisture content – CDM Smith Boston Geotechnical Laboratory
- Bulk density – CDM Smith Boston Geotechnical Laboratory

6.1 TAL Metals in Mixtures

Stabilized and solidified materials were disaggregated to less than 2 millimeters (mm) using a mortar and pestle and thoroughly homogenized at the DTL prior to submission to the CLP laboratory for TAL metals analysis. Table 6-2 provides the results for these analyses.

Comparison of metals concentrations in the original spiked untreated samples (SS-M-1500 and SS-H-6000 in Table 2-4) shows general good precision for most metals. Comparison of metal concentrations in the original spiked untreated samples to the treated samples is presented as relative percent difference (Table 6-3). Negative relative percent difference (RPD) values indicate that metal concentrations decreased in soil post-treatment, relative to the untreated spiked sample, while positive values indicate an increase post-treatment. As anticipated, samples treated with calcium polysulfide and calcium oxide had elevated concentrations of calcium. Samples treated with FerroBlack® were expected to have elevated concentrations of iron, however, concentrations were variable. Because the analytical variability is typically +/- 20 %, the addition of between 5 to 15 % FerroBlack® was not typically observed. As previously discussed, mercury in the un-treated spiked samples were 2,300 mg/kg in SS-M-1500 and 6,990/6910 mg/kg in SS-H-6000 (Table 2-4). For the medium level spiked and treated samples (SS-M-1500), mercury ranged from 1,790 mg/kg in SS-M-FB-CaO-5 to 4,070 mg/kg in SS-M-FB-5-OPTI (Table 6-2). For the high level spiked and treated sample (SS-H-6000), mercury ranged from 4,120 mg/kg in SS-H-FB-CaO-10-Solidified to 8,830 mg/kg in SS-H-FB-15-Solidified.

6.2 Synthetic Precipitation Leaching Procedure (SPLP)

SPLP leaching was performed at the DTL using a modified procedure (SOP 1-4) and analysis of the leachates was performed by CLP laboratory. The modification to the published procedure changed the solid to water ratio of 1:20 (grams per milliliter [g/mL]) to 1:2 (g/mL). This modification was performed to better represent potential leaching in the field. Table 6-1 shows the sample treatments selected for SPLP testing and includes the un-treated spiked solids (SS-H-6000 and SS-M-1500). Table 6-4 provides the mass of soil and water used to create the leachate as well as measurement parameters including pH, oxidation reduction potential (ORP) and conductivity performed by at the DTL. Table 6-5 provides results of the RCRA metals analysis performed by Chemtech Consulting Group. The stabilized and solidified samples were disaggregated to less than 2 mm prior to leaching. All stabilized materials and the original spiked samples tested were less than 2 mm prior to leaching. The untreated spiked samples had leachable quantities of mercury at 406 µg/L in SS-M-1500 and 408 µg/L in SS-H-6000 (a majority of the mercury contained in these samples was insoluble elemental mercury). For the spiked soils treated with elemental sulfur (SS-H-6000 soil spiked at 6,000 mg/kg with elemental mercury), total mercury was leached between 125 (5 percent sulfur) and 347 µg/L (25 percent sulfur). The four spiked soils that were treated with calcium polysulfide (three from SS-H-6000 and one from SS-M-1500) had mercury detected from 1,090 µg/L in SS-M-CPx-5 to 285,000 µg/L in SS-H-CPx-10-OPTI. The four spiked soils treated with FerroBlack® (three from SS-M-1500 and one from SS-H-6000) had mercury measured in the SPLP leachate between 57 µg/L in SS-M-FB-CaO-5 and

10,900 µg/L in SS-H-FB-15-OPTI. The three treated and solidified samples (all SS-H-6000) contained mercury in the SPLP leachate between 1.4 µg/L in SS-H-CPx-10-Solidified and 46,600 µg/L in SS-H-FB-CaO-10-Solidified.

The SS-H-6000 spiked sample (6,000 mg/kg) SPLP results showed the lowest mercury concentration (1.4 µg/L) in the solidified sample that was stabilized with calcium polysulfide at 10 percent. The sample that was not solidified but stabilized with calcium polysulfide at 10 percent had the highest SPLP mercury concentration (285,000 µg/L). The samples treated with sulfur had lower mercury concentrations in the SPLP leachate (125 µg/L in SS-H-S-5 and 347 in SS-H-S-25) when compared to all other SS-H treated sample results.

For SS-M spiked sample (1,500 mg/kg) SPLP results showed the lowest mercury concentrations (57 µg/L) for the sample that was stabilized with calcium oxide and 5 percent FerroBlack®.

Overall, nine of the 13 samples that were stabilized or stabilized and solidified had higher mercury concentrations in the SPLP leachate than in the SPLP leachate from the untreated spiked samples. This result is probably due of formation of mercury-sulfide complex when excess sulfide is present. Such mercury-sulfide complexes are more soluble (-log K_{sp} values of -5 to -10) when compared to mercury sulfide (-log K_{sp} values of -53 to -54).

A comparison of the elemental concentrations in the SPLP leachates is presented as relative percent difference (Table 6-6). For both SS-M and SS-H spiked and treated mixtures, significant increases in other elemental concentrations in the SPLP leachates were not observed except for chromium, lead, and arsenic in a few treatments. Chromium increased in the three solidified samples as well as several other treated samples (SS-H-CPx-10-OPTI, SS-H-S-5, SS-M-CPx-5, SS-M-FB-5, and SS-M-FB-CaO-5). Arsenic was elevated in SS-M-CPx-5 and SS-M-FB-CaO-5 by 57 percent and 60 percent, respectively, relative to the untreated spiked sample. Increases in lead were observed in SS-H-CPx-10-OPTI and SS-M-FB-CaO-5.

6.3 Regulatory Classification Analysis

To determine regulatory and safety evaluation during full scale in-situ stabilization the corrosivity and reactivity of selected mixtures were measured at the DTL. The results for these measurements are presented in Table 6-7. All measurements of H₂S gas were below the detection limit and the pH values ranged from 7.72 to 12.87.

6.4 Semi-Dynamic Leaching (SDL)

To better represent actual leaching in the field, SDL leaching was performed at the DTL using a modified SW-846 method 1315 and ASTM method 1308 (SOP 1-4). The solidified sample was immersed in synthetic rainwater (SPLP water) in a polytetrafluoroethylene (i.e., Teflon) container. The solidified materials surface area to water ratio was 1:9 (square centimeters per milliliter). The leachate was removed from the vessel and replaced with fresh SPLP water at the following time intervals: 2 hours, 24 hours, 48 hours, 72 hours, 8 days, 14 days, 21 days, 28 days and 42 days. Table 6-8 provides the solidified sample treatments selected for SDL testing and includes the dimensions and surface areas of each core leached. Table 6-9 provides the mass of water used to create the leachate at each exchange interval as well as measurement parameters

including pH, ORP, and conductivity performed at the DTL during this procedure. Table 6-10 provides results of the dissolved mercury analysis performed by Chemtech Consulting Group.

Dissolved mercury results for SS-H-CPx-10-Solidified ranged from 1.2 µg/L at 21 days to 209 µg/L at 24 hours. For sample SS-H-FB-15-Solidified, dissolved mercury ranged from 6.4 µg/L at 28 days to 231 µg/L at 2 hours. For sample SS-H-CaO-FB-10-Solidified, dissolved mercury ranged from 12 µg/L at 28 days to 329 µg/L at 2 hours.

Overall the dissolved mercury results from SS-H-CPx-10-Solidified sample were very low during the last five leaching times (8 to 42 days) ranging from 0.88 to 2.9 µg/L. Evaluation of the SDL results (see Appendix B) indicate that the initial higher concentrations are the result of surface wash off while the later lower concentrations are the result of depletion and/or dissolution of the mercury from the stabilized/solidified samples. Based on the SDL tests, the estimated mercury concentrations at the interface of water (e.g., groundwater) and the stabilized/solidified soil would be approximately 3 to 4 µg/L over the time period of 2 to 10 years. These concentrations would decrease away from the water/soil interface as a result of additional mixing with the water.

6.5 Mercury Speciation in Mixtures (Sub-Contract Laboratory)

Mercury speciation analysis was performed on three stabilized/solidified samples and ten stabilized materials by Brooks Applied Labs (BAL) in Bothel, Washington. In this method, mercury is extracted from a sample into five different solutions that can be broadly linked to types of mercury compounds. In addition, volatile elemental mercury was analyzed (F0). The extractants used were: deionized water (F1), hydrochloric acid/acetic acid at pH 2 (F2), 1M potassium hydroxide solution (F3), 12M nitric acid (F4), and aqua regia (F5). All samples were analyzed for Hg by EPA Method 1631. The primary fractions of interest are the F4 fraction in which elemental mercury is extracted and the F5 fraction which contains the mercury sulfide fraction. Results for these analyses are presented in Table 6-11. Table 6-12 presents a comparison of mercury speciation results performed by BAL versus the results produced by the CDM Smith DTL following SOP 1-3. Laboratory reports for the analyses completed by BAL can be found in Appendix C.

As shown in Table 6-11, the total mercury concentrations were higher than the sum of the fractions for 10 of the 13 samples. In five of the samples, the total concentrations were significantly higher with relative percent difference (RPD) values ranging from 33 percent to 96 percent. For SS-H-6000 treated samples, the total mercury ranged from 4,120 mg/kg to 8,830 mg/kg while the sum of the fractions ranged from 3,550 mg/kg to 6,528 mg/kg. For SS-M-1500 treated samples, the total mercury ranged from 1,790 mg/kg to 4,070 mg/kg while the sum of the fractions ranged from 1,424 mg/kg to 2,106 mg/kg. For all treated samples, the elemental mercury fraction F4, ranged from 209 mg/kg to 3,920 mg/kg while the percent of F4 (based on the sum of the fractions) ranged from 6.5 percent to 70 percent. For all treated samples, the mercury sulfide fraction F5, ranged from 842 mg/kg to 5,970 mg/kg while the percent of F5 ranged from 29 percent to 92 percent.

Table 6-12 compares the elemental mercury and mercury sulfides concentrations between the mercury speciation results by BAL and the DTL results. Based on elemental mercury, the RPD

differences between BAL and DTL ranged from -74 percent to 167 percent. Negative RPD values (2 values, -72 percent and 74 percent) had higher elemental mercury concentrations measured by the DTL compared to BAL. The positive RPD values ranging from 15 percent to 167 percent (9 samples) had lower elemental concentrations measured by DTL compared to BAL. Based on mercury sulfides, the RPD differences between BAL and DTL ranged from -115 percent to 117 percent. The negative RPD values (six values) had higher mercury sulfides concentrations measured by DTL compared to BAL. The positive PRD values (five values) had lower mercury sulfide concentrations measured by DTL compared to BAL. Overall, DTL typically had lower elemental mercury concentrations and higher mercury sulfide concentrations compared to BAL speciation results. When comparing the percent conversion of elemental mercury to mercury sulfide, the percent conversion compared well for SS-H-CPx-10, SS-M-CPx-5 and SS-M-FB-CaO-5 (typically greater than 80 percent conversion for both BAL and DTL results). Overall, the treated samples did not exhibit as much conversion of elemental mercury to mercury sulfide as shown by the DTL results when compared to the BAL results. However as previously discussed and shown on Table 6-11, the BAL method had poor mass balance (total mercury compared to the sum of the fractions) in many samples. Also, as discussed below in Section 8, the BAL results did not compare well with known standards. As a result, all BAL data have been qualified as estimated (J values).

6.6 Geotechnical Parameters

Geotechnical parameters including moisture content, bulk density, hydraulic conductivity (ASTM D504) and unconfined compressive strength (ASTM C39M) were performed at the CDM Smith Geotechnical Laboratory in Boston, MA. Results for these analyses are provided in Table 6-13. The unconfined compressive strength ranged from 113.5 pounds per square inch (psi) in SS-H-FB-15-Solidified to 610.1 psi in SS-H-CPx-10-Solidified. Hydraulic conductivity ranged from 2.62-06 centimeter per second (cm/sec) in SS-H-CPx-10-Solidified to 1.06-05 cm/sec in SS-H-FB-15-Solidified. Laboratory reports for these analyses can be found in Appendix D.

As discussed in Section 1.3, it was expected that the addition of cement and bentonite will increase strength and reduce hydraulic conductivity. Although the unconfined compressive strength and hydraulic conductivity was not measured on non-solidified samples, the anticipated hydraulic conductivity would be much higher. Overall, solidification decreases potential leaching compared to the stabilized material without solidification.

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Section 7

Data Quality Summary

The CDM Smith DTL data and the mercury speciation data were reviewed and validated by a qualified data validator that was not involved in the analyses of the samples. The Region 2 DESA/Hazardous Waste Support Branch (HWSB)/Hazardous Waste Support Section (HWSS) and their contractors validated all CLP data analyzed for mercury and metals. This included seven data packages validated for metals and total mercury.

A summary of the data quality for the treatability study data including results produced by DTL, BAL and the CLP laboratories is provided below. The Bench Scale Study Data Usability Assessment Report is included in Appendix E.

7.1 CDM Smith DTL Data Quality Summary

CDM Smith DTL performed mercury analyses on treatability study samples following the procedures and protocols discussed in Section 3.3.1 of this report and SOP 1-3. Samples were analyzed three to five times each. Results reported are the average of all multiple analyses. Sample concentrations were quantified using known mercury concentrations from NIST standard 2711 and 2710 and blanks spikes (sand spiked with a known concentration of elemental mercury). Cinnabar standards were also analyzed to determine the different forms of mercury quantified.

The NIST standards and blank spike results were within acceptable criteria. These standards had recoveries between 75 and 125% of the known concentrations for mercury. Laboratory duplicate samples were also analyzed. The RSD were calculated and generally below 25% RSD criteria. Laboratory blanks (sand blanks) did not contain measurable quantities of mercury.

The CDM Smith DTL sample results are considered representative of the elemental mercury and mercury sulfide sample concentrations. The protocols established and followed during analyses provide sufficient precision and accuracy to provide defensible and useable data to achieve project objectives.

7.2 Brooks Applied Laboratory Data Quality Summary

Brooks Applied Laboratory analyzed confirmation samples for mercury and mercury speciation using an in-house sequential extraction SOP followed by analysis of the extracts for mercury using EPA Method 1631. The mercury speciation compounds were qualified as estimated (J) for all confirmation samples based on laboratory duplicate relative percent differences and standard reference material percent recoveries. One sample result for F2 (weak acid-soluble) mercury was also qualified as non-detect based on laboratory preparation blank criteria.

The laboratory reported that the concentration used for the matrix spike and matrix spike duplicate analysis was not sufficient for proper recoveries. Therefore, the results are not valid indicators of data quality. The RPD results for these samples were not able to be evaluated.

As previously discussed, the laboratory also reported poor RPD results for six of the samples when comparing the sum of the sequential extraction fractions to the total mercury results for each sample. The % RPD ranged from 33 to 96 % for five of the treated spiked soils (positive RPD results from total mercury being greater than the sum of the fractions) and was -55 % for SS-M-1500 spiked soil (without treatment). The 5-step procedure followed by the laboratory is not designed to provide total values for mercury in the sample but to show how extractable the mercury is from the sample in each fraction. The laboratory noted it is not uncommon for the total values to not perfectly match the sum of the species.

The case narrative for the data package stated that there are no commercially available certified reference materials for the various mercury species. The standard reference materials (SRM) they use for elemental Hg, HgS, and HgCl₂ were produced by Studio Geochemica (SGC) and are not officially certified for any analyte. The laboratory stated SGC provided expected concentrations for the total mercury and each fraction and that they have been able to confirm those concentrations during analysis.

The laboratory stated that the SRM % recovery (R) results were outside of the expected results. They state that because no official control limits have been established for these SRMs and the speciation procedure, that these recoveries that are outside of the expected results are not indicative of poor data quality. They also noted they have noticed a shift in some of the recoveries due to the SRM degradation. No new SRMs were acquired or used by the laboratory for these samples. As discussed above, CDM Smith did qualify results associated with the SRMs that were outside of expected criteria as these results should be considered as estimate.

In addition, and as previously discussed, the original soil samples spiked with elemental mercury, SS-M-1500 and SS-H-6000, showed only 26.6 % of the mercury in the F4 fraction (elemental mercury) for SS-M-1500 and only 46.6/48.8 % (duplicate samples) of the mercury in the F4 fraction for SS-H-6000. According to the DTL original (before spiking) results for the composite soil samples and the amount of elemental mercury added, these values for elemental mercury (fraction F4) should have been 81 % and 96 %, respectively for SS-M-1500 and SS-H-6000.

7.3 CLP Laboratory Data Quality Summary

Metal results analyzed by CLP laboratory had applicable results qualified as estimated based on matrix spike recoveries, field duplicate relative percent differences, and interference check sample results. Some sample results were also qualified as non-detect based on laboratory blank criteria. There were also several metals non-detect results that were qualified as rejected based on interference check standard criteria.

7.4 Summary

The CDM Smith treatability study data are usable as reported. The data results are considered defensible based on method and laboratory procedures and are usable for project decisions.

The Brooks Applied Laboratory sample results were all qualified as estimated based on various quality control parameters. The laboratory also reported in their case narrative that quantified results for both samples and the standard reference materials “can be somewhat variable and therefore the method is not completely effective for determining the concentrations of individual

fractions in each sample.” The laboratory did report that the method has been shown to be “effective as a qualitative assessment of the relative percentages of each mercury fraction.” Based on professional judgement and past experience with this laboratory for this same method, the confirmation sample results are considered to be useful only for a general comparison of the sample results compared to the DTL treatability study results. However, the treatability study results from the DTL are considered more reliable and defensible due to the stringent method procedures performed and followed for sample analyses.

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Section 8

Conclusions and Recommendations

The following conclusions and recommendations are made based on results of the bench scale treatability study:

- Elemental sulfur was not effective in converting elemental mercury to mercury sulfides (by methods simulating in situ or ex situ mixing).
- Both FerroBlack® and calcium polysulfide were effective in converting elemental mercury to mercury sulfides when simulating in situ mixing with augers (up to 98 percent conversion using FerroBlack® and up to 94 percent conversion using calcium polysulfide). Based on the relative costs of both stabilization compounds, calcium polysulfide is the most cost-effective and is recommended to be used if a pilot study is performed.
- Mixing was most effective when water was added to the stabilization agent to achieve a more homogeneous distribution in the soil that resulted in a better reaction with the elemental mercury.
- Dosing of the reactive sulfide would need to be precise to prevent formation of mercury-sulfide complexes, which are more soluble than elemental mercury. To achieve this precise dosing, an extensive field program would need to be implemented during remedy implementation to define the elemental mercury concentrations at any given treatment location, both vertically and horizontally. Elemental mercury concentrations are likely highly variable. This high level of variability would make effective implementation of stabilization, without the formation of mercury-sulfide complexes, extremely difficult. Given this issue, stabilization with reactive sulfides would likely result in increased mercury solubility beyond current conditions.
- Based on leaching tests using synthetic precipitation (modified SPLP test on samples with grain size less than 2 mm), significant increase in leachate mercury concentrations were observed in 9 of the 13 samples stabilized with FerroBlack® and calcium polysulfide when compared to the unstabilized samples. Also, 2 of the 3 solidified/stabilized samples showed large increases in leachate mercury concentrations. Only two out of 13 samples showed a significant decrease in leachate mercury concentrations.
- Based on leaching tests conducted on intact stabilized and solidified samples using semi-dynamic leaching (SDL) tests, which may be more representative of actual field conditions than the SPLP, much lower mercury concentrations were observed in the leachate than in the SPLP tests. Based on the SDL tests, the estimated mercury concentrations at the interface of water (e.g., groundwater) and the stabilized/solidified soil would be approximately 3 to 4 µg/L over the time period of 2 to 10 years. No SDL tests were performed on a solidified only (not stabilized) sample, so it is uncertain how effective stabilization was at reducing long-term leachability.

- If the project moves forward with a pilot study, a method similar to or the same as performed in the DTL for elemental mercury and mercury sulfides (SOP 1-3 as modified) should be used in the field to document conversion of the elemental mercury to mercury sulfides. Confirmation samples should be sent to a commercial laboratory for sequential extraction and analyses (speciation); however, improvement of the procedure by BAL should be documented or an alternate laboratory should be selected.

Section 9

References

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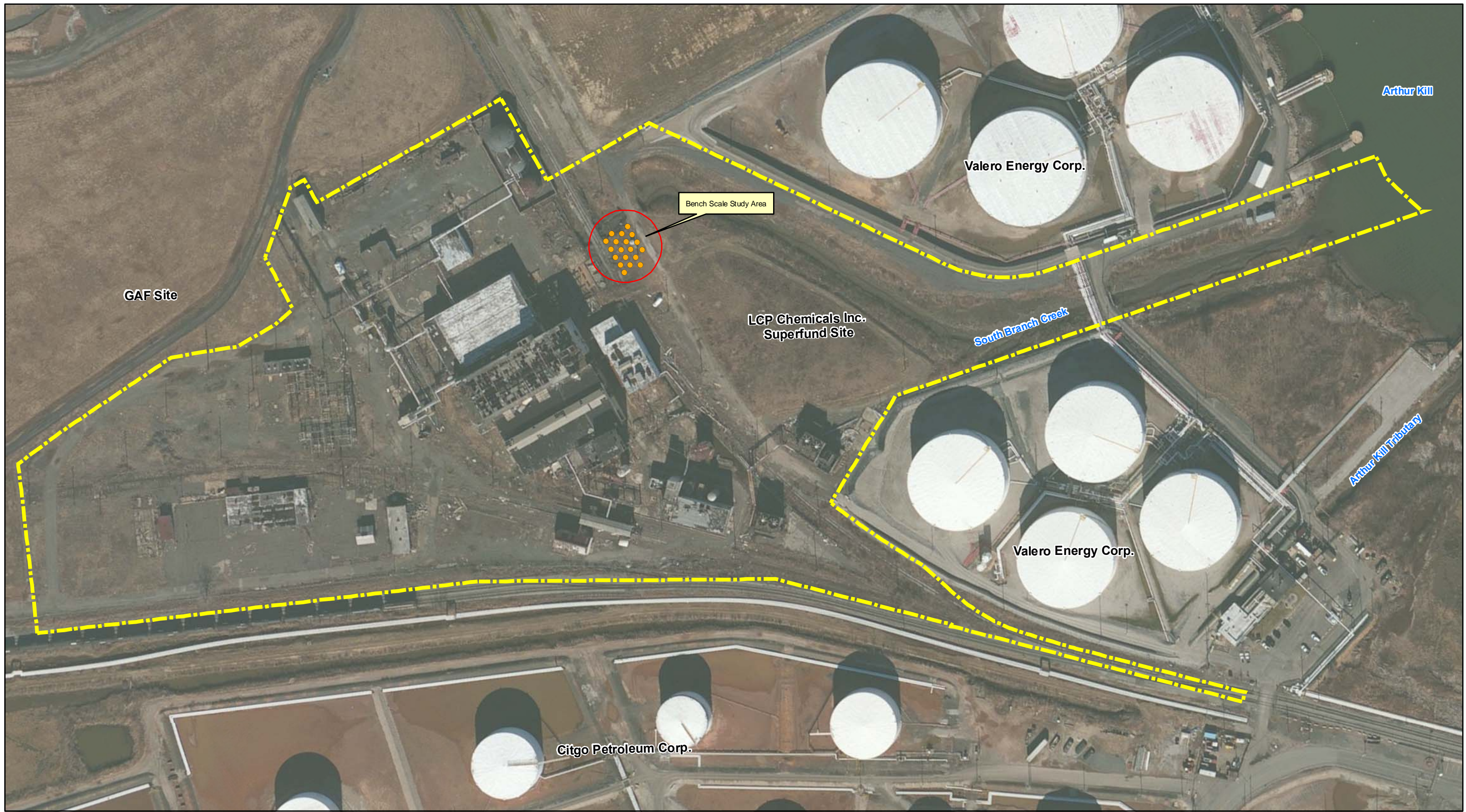
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United States Environmental Protection Agency (USEPA). 2014. Record of Decision. LCP Chemicals Inc., Superfund Site, Linden, Union County, New Jersey.

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Figures



Legend

- Bench Scale Area Borehole
- Site Boundary

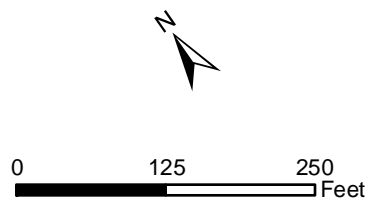


Figure 2-1
Bench Scale Study Area
Pre-Design Investigation
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

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Tables

Table 2-1 Metals in Spiked and Un-Spiked Results (Subcontract Laboratory)

Analyte	SS-M (mg/kg)		SS-H (mg/kg)		SS-H-dup (mg/kg)		SS-M-1500 (mg/kg)		SS-H-6000 (mg/kg)		SS-H-6000-dup (mg/kg)	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Aluminum	5220		5150		4700		9220		8270		8300	
Antimony	0.44	J	1.2	J	1.2	J	0.66	J	1.7	J	1.2	J
Arsenic	29		245		209		33		281		217	J
Barium	460		588		526		912		971		1030	J
Beryllium	0.35	J	1.3	J	0.36	J	0.54	U	0.55	U	0.55	U
Cadmium	1.9		5.1		3.7		1.2		3.7		3.5	
Calcium	25000		33400		31800		32800		38700		40000	
Chromium	20		48		42		29		58		57	
Cobalt	3.9	J	3.9	J	3.3	J	5.4	U	5.8		5.5	U
Copper	37		130		79		44		99		101	
Iron	13900		14300		13000		15800		16200		18500	
Lead	46		110		105		62		124		122	
Magnesium	3370		3640		3380		4310		4400		4360	
Manganese	123		124		110		146		143		142	
Mercury	NM		NM		NM		2300	J	6990	J	6910	J
Nickel	9.5		9.4		7.4		13		12	J	23	J
Potassium	790		635		597		1840		1420		1430	
Selenium	2.8	R	2.8	R	2.8	R	3.8	U	3.8	U	3.8	U
Silver	0.79	U	0.80	U	0.79	U	1.1	U	1.1	U	1.1	U
Sodium	2170		1560		1390		2380		1710		1680	
Thallium	2.0	U	2.0	U	2.00	U	2.7	U	2.7	U	2.7	U
Vanadium	16		18		16		23		26		25	
Zinc	99		362	J	189	J	176		376		318	

Notes:

RPD = relative percent difference

Q = qualifier

mg/kg = milligram/kilogram

J = the result is an estimated quantity

U = not detected above the level of the reported sample
quantitation limit

R = the result was rejected

NM = not measured

Table 2-2 Mercury Speciation in Spiked Sample Results (Subcontract Laboratory)

Fraction	General Description	SS-H-6000 (mg/kg)		SS-H-6000-dup (mg/kg)		SS-M-1500 (mg/kg)	
		Result	Q	Result	Q	Result	Q
F0	Volatile Elemental Mercury	17	J	21	J	8	J
	Fraction % of Total	0.3%		0.3%		0.2%	
F1	Water Soluble Mercury	24	J	31	J	13	J
	Fraction % of Total	0.4%		0.4%		0.3%	
F2	Weak Acid-Soluble Mercury	280	J	335	J	5.66	J
	Fraction % of Total	4.5%		4.3%		0.1%	
F3	Organo-Complexed Mercury	405	J	493	J	20.3	J
	Fraction % of Total	6.5%		6.3%		0.5%	
F4	Elemental Mercury	2940	J	3820	J	1080	J
	Fraction % of Total	46.8%		48.8%		26.6%	
F5	Mineral-Bound Mercury	2610	J	3130	J	2940	J
	Fraction % of Total	41.6%		40.0%		72.3%	
Sum of Fractions		6276		7830		4066	
Mercury, Total		6990	J	6910	J	2300	J
RPD (Total vs. Sum of Fractions)		11%		-12%		-55%	

	General Description	Extractant	Typical Species
F0	Volatile Hg	DI water	Gaseous elemental Hg
F1	Water-soluble Hg	DI water	HgCl ₂ ; (HgO); (HgSO ₄)
F2	Weak acid-soluble Hg	pH 2 HCl/HOAc	HgO; (HgSO ₄); (HgAu)
F3	Organo-complexed Hg	1 M KOH	Hg-humic acid complexes; CH ₃ Hg; Hg ₂ Cl ₂
F4	Elemental Hg	12 M HNO ₃	Elemental Hg; Hg ₂ Cl ₂
F5	Mineral-bound Hg	Aqua Regia	HgS, m-HgS, HgSe, HgAu, amalgams

Notes:

RPD = relative percent difference

Q = qualifier

mg/kg = milligram/kilogram

J = the result is an estimated quantity

U = not detected above the level of the reported sample quantitation limit

Table 3-1 Sample Identification Descriptions

Sample ID‡	Soil*	Additive	Target Additive Concentration†	Other Sample Descriptions
SS-M-S-5	SS-M	Sulfur	5%	-
SS-M-S-12.5	SS-M	Sulfur	12.5%	-
SS-M-S-25	SS-M	Sulfur	25%	-
SS-H-S-5	SS-H	Sulfur	5%	-
SS-H-S-25	SS-H	Sulfur	25%	-
SS-M-S-1000-Mill	SS-M	Sulfur	1000 mg/kg	Mixed with rotary ball mill
SS-M-S-1500-Mill	SS-M	Sulfur	1500 mg/kg	Mixed with rotary ball mill
SS-M-S-2000-Mill	SS-M	Sulfur	2000 mg/kg	Mixed with rotary ball mill
SS-H-S-4000-Mill	SS-H	Sulfur	4000 mg/kg	Mixed with rotary ball mill
SS-H-S-6000-Mill	SS-H	Sulfur	6000 mg/kg	Mixed with rotary ball mill
SS-H-S-8000-Mill	SS-H	Sulfur	8000 mg/kg	Mixed with rotary ball mill
SS-M-CPx-1.5	SS-M	Calcium polysulfide	1.5%	-
SS-M-CPx-3	SS-M	Calcium polysulfide	3%	-
SS-M-CPx-5	SS-M	Calcium polysulfide	5%	-
SS-M-CPx-5-OPTI	SS-H	Calcium polysulfide	5%	Mixing optimized, water added
SS-H-CPx-2	SS-H	Calcium polysulfide	2%	-
SS-H-CPx-6	SS-H	Calcium polysulfide	6%	-
SS-H-CPx-10	SS-H	Calcium polysulfide	10%	-
SS-H-CPx-10-OPTI	SS-H	Calcium polysulfide	10%	Mixing optimized, water added
SS-M-FB-2	SS-M	FerroBlack®	2%	-
SS-M-FB-5	SS-M	FerroBlack®	5%	-
SS-M-FB-10	SS-M	FerroBlack®	10%	-
SS-M-FB-CaO-5	SS-H	FerroBlack®	5%	10% Calcium oxide added
SS-M-FB-10-OPTI	SS-H	FerroBlack®	10%	Mixing optimized, water added
SS-H-FB-5	SS-H	FerroBlack®	5%	-
SS-H-FB-10	SS-H	FerroBlack®	10%	-
SS-H-FB-15	SS-H	FerroBlack®	15%	-
SS-H-FB-CaO-10	SS-H	FerroBlack®	10%	10% Calcium oxide added
SS-H-FB-15-OPTI	SS-H	FerroBlack®	15%	Mixing optimized, water added

Notes:

‡S = sulfur, CPx = calcium polysulfide, FB = FerroBlack®, CaO = Calcium oxide, OPTI = mixing optimized, Mill = mixed with rotary ball mill

*SS = spiked soil; SS-M = medium concentration of 1500 mg/kg; SS-H = high concentration of 6000 mg/kg.

†Weight percent of additive based on soil mass

ID = identification

% = percent

mg/kg = milligram per kilogram

Table 3-2 Mass of Soil and Additives for Elemental Sulfur using Auger Mixing

Sample ID	Soil	Approximate Hg Concentration (mg/kg)	Target Sulfur Concentration* (%)	Mass Soil (g)	Sulfur Mass Added (g)
SS-M-S-5	SS-M	1500	5	250	12.5
SS-M-S-12.5	SS-M	1500	12.5	250	31.3
SS-M-S-25	SS-M	1500	25	250	62.5
SS-H-S-5	SS-H	6000	5	250	12.5
SS-H-S-25	SS-H	6000	25	250	62.5

Notes:

*Weight percent concentration based on soil mass

ID = identification

mg/kg = milligram per kilogram

% = percent

g = gram

Table 3-3 Mass of Soil and Additives for Elemental Sulfur using Rotary Ball Mill

Sample ID	Approximate Hg Concentration (mg/kg)	Target Sulfur Concentration (mg/kg)	Mass Soil (g)	Sulfur Mass Added (g)
SS-M-S-1000-Mill	1500	1000	250	256
SS-M-S-1500-Mill	1500	1500	250	375
SS-M-S-2000-Mill	1500	2000	250	502
SS-H-S-4000-Mill	6000	4000	250	1000
SS-H-S-6000-Mill	6000	6000	250	1502
SS-H-S-8000-Mill	6000	8000	250	2002

Notes:

ID = identification

mg/kg = milligram per kilogram

g = gram

Table 3-4 Mass of Soil and Additives for Calcium Polysulfide using Auger Mixing

Sample ID	Approximate Hg Concentration (mg/kg)	Target Calcium Polysulfide Concentration* (%)	Mass Soil (g)	Mass Calcium Polysulfide Added (g)	Mass Water Added (g)
SS-M-CPx-1.5	1500	1.5	250	3.8	-
SS-M-CPx-3	1500	3	250	7.5	-
SS-M-CPx-5	1500	5	250	12.5	-
SS-M-CPx-5-OPTI	1500	5	250	12.5	12.5
SS-H-CPx-2	6000	2	250	5.1	-
SS-H-CPx-6	6000	6	250	15	-
SS-H-CPx-10	6000	10	250	25.1	-
SS-H-CPx-10-OPTI	6000	10	250	25	25

Notes:

*Weight percent concentration based on soil mass

ID = identification

mg/kg = milligram per kilogram

% = percent

g = gram

Table 3-5 Mass of Soil and Additives for Ferroblack® using Auger Mixing

Sample ID	Approximate Hg Concentration (mg/kg)	Target FerroBlack® Concentration* (%)	Mass Soil (g)	Mass FerroBlack® Added (g)	Mass Calcium Oxide Added (g)	Mass Water Added (g)
SS-M-FB-2	1500	2	250	5	-	-
SS-M-FB-5	1500	5	250	12.5	-	-
SS-M-FB-10	1500	10	250	25	-	-
SS-M-FB-CaO-5	1500	5	250	12.5	25	
SS-M-FB-10-OPTI	1500	10	250	25	-	25
SS-H-FB-5	6000	5	250	12.5	-	-
SS-H-FB-10	6000	10	250	25	-	-
SS-H-FB-15	6000	15	250	37.5	-	-
SS-H-FB-CaO-10	6000	10	250	25	25	
SS-H-FB-15-OPTI	6000	15	250	37.5	-	37.5

Notes:

*Weight percent concentration based on soil mass

ID = identification

mg/kg = milligram per kilogram

% = percent

g = gram

Table 4-1 Results of Elemental Sulfur using Auger Mixing

Sample ID	Pre-Treatment		Post-Treatment		Conversion based on Hg (%)	Conversion based on HgS (%)
	Elemental Hg (mg/kg)	HgS (mg/kg)	Elemental Hg (mg/kg)	HgS (mg/kg)		
SS-H-S-5	4190	368	2686	611	35.9	5.8
SS-H-S-25	4250	511	1845	1320	56.6	19.0
SS-M-S-5	1244	368	1007	941	19.1	46.1
SS-M-S-12.5	1100	268	848	1073	22.9	73.2
SS-M-S-25	1204	204	995	1017	17.4	67.5

Notes:

ID = identification

Hg = elemental mercury

HgS = mercuric sulfide

mg/kg = milligram per kilogram

% = percent

Table 4-2 Results of Elemental Sulfur using Rotary Ball Mill

Sample ID	Pre-Treatment		Post-Treatment		Conversion based on Hg (%)	Conversion based on HgS (%)
	Elemental Hg (mg/kg)	HgS (mg/kg)	Elemental Hg (mg/kg)	HgS (mg/kg)		
SS-H-S-4000-Mill	4610	327	4065	427	11.8	2.2
SS-H-S-6000-Mill	4238	368	3103	502	26.8	3.2
SS-H-S-8000-Mill	4154	302	3045	451	26.7	3.6
SS-M-S-1000-Mill	1179	428	865	508	26.6	6.8
SS-M-S-1500-Mill	1202	299	822	399	31.6	8.3
SS-M-S-2000-Mill	1268	317	960	406	24.3	7.0

Notes:

ID = identification

Hg = elemental mercury

HgS = mercuric sulfide

mg/kg = milligram per kilogram

% = percent

Table 4-3 Results of Calcium Polysulfide using Auger Mixing (Optimization Included)

Sample ID	Pre-Treatment		Post-Treatment		Conversion based on Hg (%)	Conversion based on HgS (%)
	Elemental Hg (mg/kg)	HgS (mg/kg)	Elemental Hg (mg/kg)	HgS (mg/kg)		
SS-H-CPx-2	4200	162	2230	1180	46.9	24.2
SS-H-CPx-6	4100	70	787	1169	80.8	26.8
SS-H-CPx-10	4210	412	895	2145	78.7	41.2
SS-H-CPx-10-OPTI	4176	407	276	3756	93.4	80.2
SS-M-CPx-1.5	947	353	221	529	76.7	18.6
SS-M-CPx-3	1003	215	269	644	73.2	42.8
SS-M-CPx-5	1110	259	94	981	91.5	65.0
SS-M-CPx-5-OPTI	1205	413	188	1541	84.4	93.6

Notes:

ID = identification

Hg = elemental mercury

HgS = mercuric sulfide

mg/kg = milligram per kilogram

% = percent

Table 4-4 Results of FerroBlack® using Auger Mixing

Sample ID	Pre-Treatment		Post-Treatment		Conversion based on Hg (%)	Conversion based on HgS (%)
	Elemental Hg (mg/kg)	HgS (mg/kg)	Elemental Hg (mg/kg)	HgS (mg/kg)		
SS-H-FB-5	3112	302	1113	1898	64.2	51.3
SS-H-FB-10	3454	328	1296	3709	62.5	97.9
SS-H-FB-15	3847	401	1095	3109	71.5	70.4
SS-H-FB-15-OPTI	3992	438	321	4254	92.0	95.6
SS-H-FB-CaO-10	3822	367	228	3542	94.0	83.1
SS-M-FB-2	1223	245	597	998	51.2	61.6
SS-M-FB-5	1302	222	453	1202	65.2	75.3
SS-M-FB-5-OPTI	1265	397	90	1594	92.9	94.6
SS-M-FB-10	1205	284	400	1305	66.8	84.7
SS-M-FB-CaO-5	1322	301	205	1298	84.5	75.4

Notes:

ID = identification

Hg = elemental mercury

HgS = mercuric sulfide

mg/kg = milligram per kilogram

% = percent

Table 5-1 Samples Selected for Solidification

Sample ID	Stabilization Agent	Concentration Stabilization Agent (%)	Concentration Cement (%)	Concentration Bentonite (%)	Concentration Water (%)
SS-H-CPx-10	Calcium polysulfide	10	10	5	10
SS-H-FB-15	FerroBlack®	15	10	5	10
SS-H-FB-CaO-10	FerroBlack® with Calcium oxide	10	5	2.5	7.5
SS-M-CPx-5	Calcium polysulfide	5	10	5	7.5
SS-M-FB-10	Ferrobblack®	10	5	2.5	7.5
SS-M-FB-CaO-5	FerroBlack® with Calcium oxide	5	5	2.5	7.5

Note: Preparation of solidified samples includes the addition of water to solidify the cement and bentonite. This process is similar to the optimized stabilization procedure where water is added to enhance amendment distribution.

% = percent

Table 6-1 Stabilized and Solidified Samples Selected for Performance Testing

Sample ID	Stabilization Agent	Concentration Stabilization Agent (%)	Treatment	Performance Test			
				TAL Metals	SPLP	SDL	Geotechnical Parameters *
SS-H-CPx-10-Solidified	Calcium polysulfide	10	Stabilized and solidified	x	x	x	x
SS-H-FB-15-Solidified	FerroBlack®	15	Stabilized and solidified	x	x	x	x
SS-H-FB-CaO-10-Solidified	FerroBlack® with Calcium oxide	10	Stabilized and solidified	x	x	x	x
SS-H-CPx-10-OPTI	Calcium polysulfide	10	Optimized stabilization	x	x		
SS-H-CPx-5-OPTI	Calcium polysulfide	5	Optimized stabilization	x	x		
SS-H-FB-15-OPTI	FerroBlack®	15	Optimized stabilization	x	x		
SS-H-CPx-10	Calcium polysulfide	10	Stabilized	x	x		
SS-H-S-25	Sulfur	25	Stabilized	x	x		
SS-H-S-5	Sulfur	5	Stabilized	x	x		
SS-H-6000	none				x		
SS-H-6000-dup	none				x		
SS-M-FB-5-OPTI	FerroBlack®	5	Optimized stabilization	x	x		
SS-M-CPx-5	Calcium polysulfide	5	Stabilized	x	x		
SS-M-FB-5	FerroBlack®	5	Stabilized	x	x		
SS-M-FB-CaO-5	FerroBlack®	5	Stabilized	x	x		
SS-M-1500	none				x		

Notes:

* Geotechnical Parameters - unconfined compressive strength, hydraulic conductivity, bulk density and moisture content

% = percent

TAL = Target Analyte List

SPLP = Synthetic Precipitation Leaching Procedure

SDL = semi-dynamic leaching

Table 6-2 Metals in Solidified and Stabilized Samples

Analyte	SS-M-1500 (mg/kg)		SS-H-6000 (mg/kg)		SS-H-6000-dup (mg/kg)		SS-H-CPX-10- Solidified (mg/kg)		SS-H-FB-15- Solidified (mg/kg)		SS-H-FB-CaO-10- Solidified (mg/kg)		SS-H-S-25 (mg/kg)		SS-M-FB-5 (mg/kg)	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Aluminum	9220		8270		8300		6160		7390		6770		4370		5790	
Antimony	0.66	J	1.7	J	1.2	J	0.91	J	1.2	J	0.97	J	1.2	J	0.67	J
Arsenic	33		281		217	J	182		195		175		240		30.9	
Barium	912		971		1030	J	156		413		543		23.8		393	
Beryllium	0.54	U	0.55	U	0.55	U	0.43		0.52		0.41	J	0.37	J	0.66	
Cadmium	1.2		3.7		3.5		3.7		4.5		3.6		3.6		2.0	
Calcium	32800		38700		40000		69700		61400		56600		28400		26800	
Chromium	29		58		57		40		48		39		38		21	
Cobalt	5.4	U	5.8		5.5	U	4.1		4.3		4.0	J	3.0	J	4.6	
Copper	44		99		101		73		78		71		70		51	
Iron	15800		16200		18500		13300		19900		16200		12400		14600	
Lead	62		124		122		82		98		95		73		53	
Magnesium	4310		4400		4360		3610		3690		3630		3150		3610	
Manganese	146		143		142		216		254		250		96		135	
Mercury	2300	J	6990	J	6910	J	5340	J	8830	J	4120	J	7830	J	2740	J
Nickel	13		12	J	23	J	10		11		11		6.6		11	
Potassium	1840		1420		1430		1160		1170		1090		569		829	
Selenium	3.8	U	3.8	U	3.8	U	2.7	R	3.0	R	3.0	R	2.7	R	2.6	R
Silver	1.1	U	1.1	U	1.1	U	0.77	UJ	0.86	U	0.86	U	0.78	U	0.76	U
Sodium	2380		1710		1680		2210		9320		6760		1280		4300	
Thallium	2.7	U	2.7	U	2.7	U	1.9	U	2.2	U	2.1	U	2.0	U	1.9	U
Vanadium	23		26		25		19		22		19		15		16	
Zinc	176		376		318		250		259		267		206		179	

Notes:

mg/kg = milligrams per kilogram

Q = qualifier

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R = The analyte was analyzed for, but the presence or absence of the analyte has not been verified.

Table 6-2 Metals in Solidified and Stabilized Samples

Analyte	SS-H-CPX-10 (mg/kg)		SS-M-FB-CAO-5 (mg/kg)		SS-M-CPX-5 (mg/kg)		SS-H-CPX-10-OPTI (mg/kg)		SS-H-S-5 (mg/kg)		SS-M-CPX-5-OPTI (mg/kg)		SS-H-FB-15-OPTI (mg/kg)		SS-M-FB-5-OPTI (mg/kg)	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
Aluminum	5300		4900		4980		5050		4720		4870		5790		5020	
Antimony	1	J	1.2	J	0.51	J	1.2	J	1.3	J	0.47	J	1.3	J	0.71	J
Arsenic	240		164		28		217		200		28		189		28	
Barium	141		619		226		169		69		159		399		335	
Beryllium	0.4		0.47		0.53		0.36		0.35	J	0.35	J	0.38		0.36	
Cadmium	3.8		4.0		3.1		4.8		3.4		2.2		4.7		2.2	
Calcium	39600		97200		29100		39800		30100		31100		31100		27300	
Chromium	48		38		20		44		42		21		46		21	
Cobalt	5.1		3.3	J	3.8	J	3.7		3.4	J	3.9		3.9		4.1	
Copper	90		67		41		77		76		37		77		40	
Iron	13800		12700		12700		14200		13000		13300		21800		16900	
Lead	89		105		54		92		95		49		96		55	
Magnesium	3730		3620		3140		3800		3320		3380		3410		3360	
Manganese	185		102		126		111		103		121		149		141	
Mercury	5990	J	1790	J	1840	J	7610	J	6280	J	2830	J	4840	J	4070	J
Nickel	9.4		8.3		10		8.6		7.4		9.2		12		10	
Potassium	676		647		759		684		587		740		573		719	
Selenium	2.8	R	2.7	R	2.8	R	2.5	R	2.7	R	2.6	R	2.7	R	2.5	R
Silver	0.79	U	0.78	U	0.79	U	0.71	U	0.78	U	0.73	U	0.77	U	0.72	U
Sodium	1530		4020		2040		1470		1400		2100		8910		7720	
Thallium	2.0	U	2.0	U	2.0	U	1.8	U	2.0	U	1.8	U	1.9	U	1.8	U
Vanadium	18		16		14		17		16		15		16		15	
Zinc	211		231		222		259		215		109		321		138	

Notes:

mg/kg = milligrams per kilogram

Q = qualifier

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R = The analyte was analyzed for, but the presence or absence of the analyte has not been verified.

Table 6-3 Comparison of Metals in Samples Pre and Post Treatment

Analyte	SS-H-CPx-10-Solidified	SS-H-FB-15-Solidified	SS-H-FB-CaO-10-Solidified	SS-H-S-25	SS-M-FB-5	SS-H-CPx-10	SS-M-FB-CaO-5
	RPD	RPD	RPD	RPD	RPD	RPD	RPD
Aluminum	-26%	-11%	-18%	-47%	-37%	-36%	-47%
Antimony	-46%	-29%	-43%	-29%	2%	-41%	82%
Arsenic	-35%	-31%	-38%	-15%	-5%	-15%	405%
Barium	-84%	-57%	-44%	-98%	-57%	-85%	-32%
Beryllium	-22%	-5%	-25%	-33%	22%	-27%	-13%
Cadmium	0%	22%	-3%	-3%	67%	3%	233%
Calcium	80%	59%	46%	-27%	-18%	2%	196%
Chromium	-30%	-17%	-32%	-34%	-28%	-17%	31%
Cobalt	-29%	-26%	-31%	-48%	-15%	-12%	-39%
Copper	-26%	-22%	-28%	-29%	17%	-9%	53%
Iron	-18%	23%	0%	-23%	-8%	-15%	-20%
Lead	-34%	-21%	-23%	-41%	-14%	-29%	69%
Magnesium	-18%	-16%	-18%	-28%	-16%	-15%	-16%
Manganese	51%	78%	75%	-33%	-8%	29%	-30%
Mercury	-24%	26%	-41%	12%	19%	-14%	-22%
Nickel	-15%	-5%	-11%	-45%	-18%	-22%	-36%
Potassium	-18%	-18%	-23%	-60%	-55%	-52%	-65%
Selenium	-29%	-21%	-21%	-29%	-32%	-26%	-29%
Silver	-30%	-22%	-22%	-29%	-31%	-28%	-29%
Sodium	29%	445%	295%	-25%	81%	-11%	69%
Thallium	-30%	-19%	-22%	-26%	-30%	-26%	-26%
Vanadium	-27%	-16%	-29%	-42%	-29%	-33%	-29%
Zinc	-34%	-31%	-29%	-45%	2%	-44%	-31%

Notes:

RPD = Relative percent difference between samples pre- and post-treatment. Negative values indicate a decrease post-treatment, positive values indicate an increase.

% = percent

Table 6-3 Comparison of Metals in Samples Pre and Post Treatment

Analyte	SS-M-CPx-5	SS-H-CPx-10-OPTI	SS-H-S-5	SS-M-CPx-5-OPTI	SS-H-FB-15-OPTI	SS-M-FB-5-OPTI
	RPD	RPD	RPD	RPD	RPD	RPD
Aluminum	-46%	-39%	-43%	-47%	-30%	-46%
Antimony	-23%	-29%	-24%	-29%	-24%	8%
Arsenic	-15%	-23%	-29%	-14%	-33%	-14%
Barium	-75%	-83%	-93%	-83%	-59%	-63%
Beryllium	-2%	-35%	-36%	-35%	-31%	-33%
Cadmium	158%	30%	-8%	83%	27%	83%
Calcium	-11%	3%	-22%	-5%	-20%	-17%
Chromium	-31%	-24%	-27%	-30%	-20%	-28%
Cobalt	-30%	-36%	-41%	-28%	-33%	-24%
Copper	-7%	-23%	-24%	-15%	-23%	-9%
Iron	-20%	-12%	-20%	-16%	35%	7%
Lead	-13%	-26%	-23%	-21%	-23%	-11%
Magnesium	-27%	-14%	-25%	-22%	-23%	-22%
Manganese	-14%	-22%	-28%	-17%	4%	-3%
Mercury	-20%	9%	-10%	23%	-31%	77%
Nickel	-22%	-28%	-38%	-29%	-1%	-22%
Potassium	-59%	-52%	-59%	-60%	-60%	-61%
Selenium	-26%	-34%	-29%	-32%	-29%	-34%
Silver	-28%	-35%	-29%	-34%	-30%	-35%
Sodium	-14%	-14%	-18%	-12%	421%	224%
Thallium	-26%	-33%	-26%	-33%	-30%	-33%
Vanadium	-37%	-33%	-37%	-34%	-37%	-34%
Zinc	26%	-31%	-43%	-38%	-15%	-22%

Notes:

RPD = Relative percent difference between samples pre- and post-treatment. Negative values indicate a decrease post-treatment, positive values indicate an increase.

% = percent

Table 6-4 Synthetic Precipitation Leaching Procedure Parameters

Sample ID	Soil (g)	Leach Water (g)	Beginning Parameters			Ending Parameters		
			pH (s.u.)	ORP (mV)	Conductivity (mS/cm)	pH (s.u.)	ORP (mV)	Conductivity (mS/cm)
SS-H-CPx-10-Solidified	150.1	323.2	11.75	221.8	2.90	11.97	43.2	8.40
SS-H-FB-15-Solidified	150.1	314.8	11.72	129.8	7.40	12.05	53.1	16.30
SS-H-FB-CaO-10-Solidified	75.1	151.5	11.91	108.7	4.80	12.27	35.8	12.30
SS-H-CPx-10-OPTI	75.2	170.5	8.93	119.6	5.70	9.11	138.9	8.20
SS-H-CPx-5-OPTI	75.3	154.2	8.92	88.6	6.10	8.97	147.3	9.50
SS-H-FB-15-OPTI	75.1	152.3	8.68	103.1	15.20	8.53	103.9	>19.90
SS-H-CPx-10	76.2	153.2	7.98	115.7	5.40	7.77	113.1	9.10
SS-H-S-25	74.0	158.5	8.06	87.6	2.40	7.82	41.2	4.60
SS-H-S-5	75.0	157.4	8.03	89.4	3.20	7.79	45.8	5.20
SS-H-6000	76.0	155.4	8.05	94.1	2.90	7.82	69.5	5.30
SS-H-6000-dup	75.9	154.1	8.07	91.8	2.80	7.87	66.5	5.40
SS-M-FB-5-OPTI	75.5	156.3	8.38	110.6	12.00	8.43	115.5	>19.90
SS-M-CPx-5	75.9	153.8	7.80	104.4	6.20	7.58	112.9	8.50
SS-M-FB-5	75.2	151.7	8.09	107.0	7.60	7.87	75.2	12.50
SS-M-FB-CaO-5	76.7	157.9	12.57	-16.3	9.80	12.77	-55.0	19.20
SS-M-1500	75.1	156.4	8.33	97.5	5.10	8.03	64.8	7.60

Notes:

ID = identification

G = grams

s.u. = standard units

mV = millivolt

mS/cm = millisiemens per centimeter

Table 6-5 Synthetic Precipitation Leaching Procedure Sample Results

Sample ID	Arsenic (µg/L)		Barium (µg/L)		Cadmium (µg/L)		Chromium (µg/L)		Lead (µg/L)		Mercury (µg/L)		Selenium (µg/L)		Silver (µg/L)	
	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
SS-H-CPx-10-Solidified	9.6	J	244		5.0	U	20		10	U	1.4		8.9	J	10	UJ
SS-H-FB-15-Solidified	14		91	J	5.0	U	36		10	U	13200		15	J	10	U
SS-H-FB-CaO-10-Solidified	70		1180		5.0	U	68		14		46600		11	J	10	U
SS-H-CPx-10-OPTI	94		107	J	5.6		37		99		285000		4.7	J	10	U
SS-H-CPx-5-OPTI	22		31	J	5.0	U	1.4	J	10	U	7450		35	U	10	U
SS-H-FB-15-OPTI	22		22	J	5.0	U	3.2	J	10	U	10900		7.4	J	10	U
SS-H-CPx-10	47		87	J	8.0		1.5	J	10	U	4610		11	J	10	U
SS-H-S-25	176		54	J	5.0	U	3.7	J	10	U	347		35	U	10	U
SS-H-S-5	187		19	J	5.0	U	18		10	U	125		7.8	J	10	U
SS-H-6000	232	J	358	J	1.9	J	16	J	19		408	J	35	U	10	U
SS-H-6000-dup	180	J	21	J	1.0	J	3.5	J	10	U	29	J	3.9	J	10	U
SS-M-FB-5-OPTI	17		28	J	5.0	U	1.4	J	10	U	2700		4.3	J	10	U
SS-M-CPx-5	29		28	J	5.0	U	3.0	J	10	U	1090		35	U	10	U
SS-M-FB-5	12		70	J	5.0	U	2.1	J	10	U	3160		35	U	10	U
SS-M-FB-CaO-5	29		384		5.0	U	5.9	J	93		57		35	U	10	U
SS-M-1500	18		42	J	5.0	U	1.8	J	10	U	406		3.8	J	10	U

Notes:

µg/L = micrograms per liter

Q = qualifier

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

Table 6-6 Comparison of Synthetic Precipitation Leaching Procedure Results Pre and Post Treatment

Analyte	Arsenic	Barium	Cadmium	Chromium	Lead	Mercury	Selenium	Silver
	RPD	RPD	RPD	RPD	RPD	RPD	RPD	RPD
SS-H-CPx-10-Solidified	-96%	-32%	163%	27%	-47%	-100%	-75%	0%
SS-H-FB-15-Solidified	-94%	-74%	163%	128%	-47%	3135%	-58%	0%
SS-H-FB-CaO-10-Solidified	-70%	230%	163%	333%	-26%	11322%	-68%	0%
SS-H-CPx-10-OPTI	-59%	-70%	195%	139%	424%	69753%	-87%	0%
SS-H-CPx-5-OPTI	-91%	-91%	163%	-91%	-47%	1726%	0%	0%
SS-H-FB-15-OPTI	-91%	-94%	163%	-79%	-47%	2572%	-79%	0%
SS-H-CPx-10	-80%	-76%	321%	-90%	-47%	1030%	-68%	0%
SS-H-S-25	-24%	-85%	163%	-76%	-28%	-15%	0%	0%
SS-H-S-5	-19%	-95%	163%	15%	-47%	-69%	-78%	0%
SS-M-FB-5-OPTI	-10%	-34%	0%	-22%	0%	565%	13%	0%
SS-M-CPx-5	57%	-34%	0%	67%	0%	168%	821%	0%
SS-M-FB-5	-35%	66%	0%	17%	0%	678%	821%	0%
SS-M-FB-CaO-5	60%	812%	0%	228%	828%	-86%	821%	0%

Notes:

RPD = Relative percent difference between samples pre- and post-treatment. Negative values indicate a decrease post-treatment, positive values indicate an increase.

% = percent

Table 6-7 Corrosivity and Reactivity of Solidified and Stabilized Samples

Sample ID	Corrosivity pH (s.u.)	Reactivity H ₂ S gas* (mL/m ³)
SS-H-CPx-10-Solidified	11.36	<1
SS-H-FB-15-Solidified	11.61	<1
SS-H-FB-CaO-10-Solidified	12.18	<1
SS-H-CPx-10-OPTI	8.59	<1
SS-H-CPx-5-OPTI	8.86	<1
SS-H-FB-15-OPTI	8.37	<1
SS-H-CPX-10	7.72	<1
SS-H-S-25	8.01	<1
SS-H-S-5	7.96	<1
SS-H-6000	8.00	<1
SS-M-FB-5-OPTI	8.22	<1
SS-M-CPx-5	7.68	<1
SS-M-FB-5	8.03	<1
SS-M-FB-5	12.87	<1
SS-M-1500	8.08	<1

Notes:

*Reactivity is the measurement of hydrogen sulfide (H₂S) gas generated at pH 2.

s.u. = standard units

mL/m³ = milliliter per cubic meter

Table 6-8 Stabilized and Solidified Samples Selected for Semi-Dynamic Leaching

Sample ID	Stabilization Agent	Concentration Stabilization Agent (%)	Starting Dimensions (cm)	Starting Surface Area (cm)
SS-H-CPx-10-Solidified	Calcium Polysulfide	10	5.1 x 4.5	103
SS-H-FB-15-Solidified	FerroBlack [®]	15	4.9 x 4.5	101
SS-H-CaO-FB-10-Solidified	FerroBlack [®] with Calcium oxide	10	5.2 x 4.5	105

Notes:

% = percent

cm = centimeter

Table 6-9 Semi-Dynamic Leaching Parameters

Sample ID	Time Interval	Leach Water added (g)	Interval Ending Parameters		
			pH (s.u.)	ORP (mV)	Conductivity (mS/cm)
SS-H-CPx-10-Solidified	2 hrs	869.7	10.49	155.6	0.30
	24 hrs	858.7	11.34	108.9	1.50
	48 hrs	865.5	11.48	241.7	1.20
	72 hrs	877.5	11.27	113.6	1.70
	8 days	890.4	11.42	28.0	1.90
	14 days	886.8	11.41	17.9	1.70
	21 days	904.3	11.26	16.4	1.60
	28 days	921.6	11.30	16.1	1.40
	42 days	922.6	11.31	14.7	1.60
SS-H-FB-15-Solidified	2 hrs	876.9	10.70	152.6	1.10
	24 hrs	839.2	11.48	132.4	2.80
	48 hrs	860.6	11.46	189.4	1.60
	72 hrs	848.3	11.41	120.2	1.60
	8 days	892.2	11.49	31.5	1.60
	14 days	895.2	11.62	16.3	1.70
	21 days	920.6	11.34	4.6	1.50
	28 days	917.5	11.29	14.3	1.60
	42 days	917.3	11.32	15.8	1.60
SS-H-CaO-FB-10-Solidified	2 hrs	881.2	10.91	142.2	1.30
	24 hrs	854.1	11.55	122.8	3.00
	48 hrs	868.1	11.49	176.0	1.20
	72 hrs	869.8	11.48	115.9	1.80
	8 days	917.4	11.66	27.9	1.60
	14 days	917.6	11.39	18.2	1.80
	21 days	916.5	11.29	17.3	1.40
	28 days	912.4	11.27	10.9	1.60
	42 days	920.6	11.30	12.9	1.70

Notes:

g = gram

s.u. = standard unit

mV - millivolt

mS/cm = millisiemens per centimeter

Table 6-10 Semi-Dynamic Leaching Results

Sample ID	Time Interval	Mercury (µg/L)	
		Result	Q
SS-H-CPx-10-Solidified	2 hours	16	
	24 hours	209	
	48 hours	70	
	72 hours	8.9	
	8 days	1.3	
	14 days	0.88	
	21 days	1.2	
	28 days	2.2	
	42 days	2.9	
SS-H-FB-15-Solidified	2 hours	231	
	24 hours	5.4	
	48 hours	2.5	
	72 hours	2.7	
	8 days	3.7	
	14 days	8	
	21 days	7.6	
	28 days	6.4	
	42 days	44	
SS-H-CaO-FB-10-Solidified	2 hours	329	
	24 hours	182	
	48 hours	112	
	72 hours	57	
	8 days	24	
	14 days	19	
	21 days	46	
	28 days	12	
	42 days	124	

Notes:

µg/L = micrograms per liter

Q = qualifier

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate.

Table 6-11 Results of Mercury Speciation in Stabilized and Solidified Samples (Subcontract Laboratory)

Fraction	General Description	SS-H-CPx-10-Solidified		SS-H-FB-15-Solidified		SS-H-FB-CaO-10-Solidified		SS-H-S-25		SS-M-FB-5		SS-H-CPx-10		SS-M-FB-CaO-5	
		Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
F0	Volatile Elemental Mercury	0.17	J	301	J	336	J	7.5	J	0.23	J	22	J	2.9	J
	<i>Fraction % of Total</i>	<i>0.003%</i>		<i>4.7%</i>		<i>9.5%</i>		<i>0.14%</i>		<i>0.01%</i>		<i>0.33%</i>		<i>0.17%</i>	
F1	Water Soluble Mercury	0.03	J	420	J	354	J	8.9	J	4	J	44	J	5	J
	<i>Fraction % of Total</i>	<i>0.001%</i>		<i>6.6%</i>		<i>10%</i>		<i>0.17%</i>		<i>0.24%</i>		<i>0.68%</i>		<i>0.26%</i>	
F2	Weak Acid-Soluble Mercury	23	J	0.46	J	39	J	49.4	J	0.26	J	0.12	UJ	0.31	J
	<i>Fraction % of Total</i>	<i>0.4%</i>		<i>0.01%</i>		<i>1.1%</i>		<i>0.92%</i>		<i>0.01%</i>		<i>0.002%</i>		<i>0.018%</i>	
F3	Organo-Complexed Mercury	9.6	J	181	J	6.5	J	87	J	6.4	J	45	J	5.29	J
	<i>Fraction % of Total</i>	<i>0.18%</i>		<i>2.9%</i>		<i>0.18%</i>		<i>1.6%</i>		<i>0.37%</i>		<i>0.69%</i>		<i>0.3%</i>	
F4	Elemental Mercury	2820	J	2770	J	949	J	3230	J	528	J	423	J	209	J
	<i>Fraction % of Total</i>	<i>54%</i>		<i>44%</i>		<i>27%</i>		<i>60%</i>		<i>30%</i>		<i>6.5%</i>		<i>12%</i>	
F5	Mineral-Bound Mercury	2390	J	2670	J	1870	J	1970	J	1200	J	5970	J	1520	J
	<i>Fraction % of Total</i>	<i>46%</i>		<i>42%</i>		<i>53%</i>		<i>37%</i>		<i>69%</i>		<i>92%</i>		<i>87%</i>	
Sum of Fractions		5243		6342		3555		5352		1739		6504		1742	
Mercury, Total		5340	J	8830	J	4120	J	7830	J	2740	J	5990	J	1790	J
RPD (Total vs. Sum of Fractions)		2%		33%		15%		38%		45%		-8%		3%	

Notes:

All results are in milligram/kilogram (mg/kg)

Q = qualifier

J = the result is an estimated quantity

U = not detected above the level of the reported sample quantitation limit

UJ = not detected, the reported quantitation limit is approximate and may be inaccurate

RPD = relative percent difference

	General Description	Extractant	Typical Species
F0	Volatile Hg	DI water	Gaseous elemental Hg
F1	Water-soluble Hg	DI water	HgCl ₂ ; (HgO); (HgSO ₄)
F2	Weak acid-soluble Hg	pH 2 HCl/HOAc	HgO; (HgSO ₄); (HgAu)
F3	Organo-complexed Hg	1 M KOH	Hg-humic acid complexes; CH ₃ Hg; Hg ₂ Cl ₂
F4	Elemental Hg	12 M HNO ₃	Elemental Hg; Hg ₂ Cl ₂
F5	Mineral-bound Hg	Aqua Regia	HgS, m-HgS, HgSe, HgAu, amalgams

Table 6-11 Results of Mercury Speciation in Stabilized and Solidified Samples (Subcontract Laboratory)

Fraction	General Description	SS-M-CPx-5		SS-H-CPx-10-OPTI		SS-H-S-5		SS-M-CPx-5-OPTI		SS-H-FB-15-OPTI		SS-M-FB-CaO-5-OPTI	
		Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
F0	Volatile Elemental Mercury	75	J	262	J	15	J	0.15	J	0.61	J	0.07	J
	<i>Fraction % of Total</i>	3.6%		4.0%		0.24%		0.01%		0.02%		0.005%	
F1	Water Soluble Mercury	10	J	304	J	11	J	0.74	J	1.1	J	0.56	J
	<i>Fraction % of Total</i>	0.49%		4.7%		0.18%		0.05%		0.03%		0.04%	
F2	Weak Acid-Soluble Mercury	0.18	J	0.37	J	79	J	0.26	J	0.87	J	0.12	J
	<i>Fraction % of Total</i>	0.008%		0.006%		1.2%		0.02%		0.02%		0.01%	
F3	Organo-Complexed Mercury	27	J	32	J	9.5	J	12	J	22	J	5.6	J
	<i>Fraction % of Total</i>	1.3%		0.5%		0.1%		0.8%		0.6%		0.4%	
F4	Elemental Mercury	363	J	3040	J	3920	J	599	J	2770	J	519	J
	<i>Fraction % of Total</i>	17%		47%		62%		41%		70%		36%	
F5	Mineral-Bound Mercury	1630	J	2890	J	2320	J	842	J	1140	J	899	J
	<i>Fraction % of Total</i>	77%		44%		37%		58%		29%		63%	
Sum of Fractions		2106		6528		6355		1454		3934		1424	
Mercury, Total		1840		7610		6280		2830		4840		4070	
RPD (Total vs. Sum of Fractions)		-13%		15%		-1%		64%		21%		96%	

Notes:

All results are in milligram/kilogram (mg/kg)

Q = qualifier

J = the result is an estimated quantity

U = not detected above the level of the reported sample quantitation limit

UJ = not detected, the reported quantitation limit is approximate and may be inaccurate

RPD = relative percent difference

	General Description	Extractant	Typical Species
F0	Volatile Hg	DI water	Gaseous elemental Hg
F1	Water-soluble Hg	DI water	HgCl ₂ ; (HgO); (HgSO ₄)
F2	Weak acid-soluble Hg	pH 2 HCl/HOAc	HgO; (HgSO ₄); (HgAu)
F3	Organo-complexed Hg	1 M KOH	Hg-humic acid complexes; CH ₃ Hg; Hg ₂ Cl ₂
F4	Elemental Hg	12 M HNO ₃	Elemental Hg; Hg ₂ Cl ₂
F5	Mineral-bound Hg	Aqua Regia	HgS, m-HgS, HgSe, HgAu, amalgams

Table 6-12 Comparison of Mercury Speciation Results (Brooks Applied Labs versus DTL)

Fraction	General Description	SS-H-S-25		SS-M-FB-5		SS-H-CPx-10-OPTI		SS-H-CPx-10		SS-M-FB-CaO-5		SS-M-CPx-5	
		Result	Q	Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
F4	Elemental Mercury BAL	3230	J	528	J	3040	J	423	J	209	J	363	J
	Elemental Mercury DTL	1845		453		276		895		453		94	
Relative Percent Difference		55		15		167		-72		-74		118	
F5	Mineral-Bound Mercury (BAL)	1970	J	1200	J	2890	J	5970	J	1520	J	1630	J
	Mercury Sulfide (DTL)	1320.00		1202.00		3756		2145		1202		981	
Relative Percent Difference		39.51		-0.17		-26		94		23		50	

Fraction	General Description	SS-H-CPx-10-OPTI		SS-H-S-5		SS-M-CPx-5-OPTI		SS-H-FB-15-OPTI		SS-M-FB-CaO-5-OPTI	
		Result	Q	Result	Q	Result	Q	Result	Q	Result	Q
F4	Elemental Mercury BAL	3040	J	3920	J	599	J	2770	J	519	J
	Elemental Mercury DTL	276		2689		188		321		90	
Relative Percent Difference		167		37		104		158		141	
F5	Mineral-Bound Mercury (BAL)	2890	J	2320	J	842	J	1140	J	899	J
	Mercury Sulfide (DTL)	3756		611		1541		4254		1594	
Relative Percent Difference		-26		117		-59		-115		-56	

Notes:

All results are in milligram/kilogram (mg/kg)

Q = qualifier

J = the result is an estimated quantity

U = not detected above the level of the reported sample quantitation limit

UJ = not detected, the reported quantitation limit is approximate and may be inaccurate

Table 6-13 Geotechnical Parameter Results

Sample ID	Moisture Content (%)	Unconfined Compressive Strength (psi)	Hydraulic Conductivity (cm/sec)	Bulk Density (pcf)
SS-H-CPx-10-Solidified	12.3	610.1	2.62E-06	89.9
SS-H-FB-15-Solidified	6.7	113.5	1.60E-05	94.1
SS-H-CaO-FB-10-Solidified	5.0	583.8	2.13E-05	84.6

Notes:

% = percent

psi = pounds per square inch

cm/sec = centimeters per second

pcf = pounds per cubic foot

A decorative graphic consisting of a vertical blue line and a horizontal blue line intersecting at the bottom-left. A blue square is located in the bottom-left corner, with its top-right corner at the intersection of the lines.

Appendices

Appendix A

Additive Comparison

Additive Comparison Table
Bench Scale Treatability Study Report
LCP Chemicals, Inc. Superfund Site
Linden, Union County, New Jersey

Stabilization Chemical	Generic Chemicals		Proprietary Chemicals		
	Calcium Polysulfide	Sodium Sulfide	Ferrous Sulfide (FerroBlack®)	Ferrous Sulfide (FerroBlack®) and Calcium Oxide	Molecular Bonding System (MBS®)
Demonstrated Effectiveness in the Literature	<ul style="list-style-type: none">• Demonstrated effective at bench, pilot and full scale testing	<ul style="list-style-type: none">• Demonstrated effective at bench scale, and in conjunction with calcium polysulfide at pilot and full scale testing	<ul style="list-style-type: none">• Demonstrated effective at bench scale testing and field pilot and large scale applications	<ul style="list-style-type: none">• Demonstrated effective at bench scale testing	<ul style="list-style-type: none">• Demonstrated effective at bench and ex-situ scale testing
Demonstrated Effectiveness in Bench Scale Tests with samples from LCP Chemicals	<ul style="list-style-type: none">• Demonstrated effective at site specific bench scale testing• Average conversion 56%• Average conversion with optimized mixing procedures 88%	<ul style="list-style-type: none">• Not tested	<ul style="list-style-type: none">• Demonstrated effective at site specific bench scale testing• Average conversion 69%• Average conversion with optimized mixing procedures 94%	<ul style="list-style-type: none">• Demonstrated effective at site specific bench scale testing• Average conversion 84%	<ul style="list-style-type: none">• Not tested
Health and Safety	<ul style="list-style-type: none">• High pH• Corrosive• Strong odor• Potential generation of H₂S gas	<ul style="list-style-type: none">• Corrosive• Prevent contact with acids• Maintain at high pH• Potential generation of H₂S gas	<ul style="list-style-type: none">• Contains sulfide• Prevent contact with acids• Maintain at high pH	<ul style="list-style-type: none">• Contains sulfide• Prevent contact with acids• Maintain at elevated pH• Monitor Hg emissions with application of calcium oxide	<ul style="list-style-type: none">• Proprietary mixture, no health and safety issues known
Physical and Chemical Properties	<ul style="list-style-type: none">• Thick orange liquid• pH of 10.5	<ul style="list-style-type: none">• Liquid-phase (60% solid dissolved in water)	<ul style="list-style-type: none">• Solid mass suspended in liquid (10 micron sized particles of FeS)	<ul style="list-style-type: none">• Ferro Black® - solid mass suspended in liquid (10 micron sized particles of FeS)• Calcium oxide - solid	<ul style="list-style-type: none">• Solid-phase• Can be wetted to ensure mixing
Unit Cost	Graus Chemicals, Remotox bulk \$1.484/gal ¹	Sodium Sulfide \$500/ton ²	REDOX Solutions LLC, Ferro Black® bulk \$0.39/lb ¹	REDOX Solutions LLC, Ferro Black® bulk \$0.39/lb ¹ Calcium Oxide \$120/ton ²	Solucorp Industries Ltd, MBS® \$30-100/waste ton ¹ (generated amount of stabilized material)
Quantity Required* (Assuming 23,600 yds ³ of soil to be treated)	Calcium Polysulfide (29%) <ul style="list-style-type: none">• minium 476 tons for 1.5% (w/w)• maximum 3176 tons for 10% (w/w)	Sodium Sulfide (60%) <ul style="list-style-type: none">• minimum 476 tons for 1.5% (w/w)• maximum 3176 tons for 10% (w/w)	FerroBlack® <ul style="list-style-type: none">• minimum 635 tons for 2% (w/w)• maximum 4764 tons for 15% (w/w)	FerroBlack® <ul style="list-style-type: none">• minimum 635 tons for 2% (w/w)• maximum 4764 tons for 15% (w/w) Calcium Oxide <ul style="list-style-type: none">• 3175 tons for 10% (w/w)	Assume 31800 waste tons of treated soil (assuming density of 1.6 g/cm ³)
Total Chemical Cost*	<ul style="list-style-type: none">• minimum \$133,560• maximum \$890,400	<ul style="list-style-type: none">• minimum \$238,170• maximum \$1,587,830	<ul style="list-style-type: none">• minimum \$495,400• maximum \$3,715,920	<ul style="list-style-type: none">• minimum \$876,480• maximum \$4,096,595	<ul style="list-style-type: none">• minimum \$952,700 (\$30/waste ton)• maximum \$3,175,660 (\$100/waste ton)
Relative Cost/kg of Hg* (assuming Hg 1500mg/kg soil)	<ul style="list-style-type: none">• minimum \$3.08• maximum \$20.53	<ul style="list-style-type: none">• minimum \$5.50• maximum \$36.67	<ul style="list-style-type: none">• minimum \$11.44• maximum \$85.80	<ul style="list-style-type: none">• minimum \$20.24• maximum \$94.60	<ul style="list-style-type: none">• minimum \$22.00• maximum \$73.33

Additive Comparison Table
Bench Scale Treatability Study Report
LCP Chemicals, Inc. Superfund Site
Linden, Union County, New Jersey

Stabilization Chemical	Generic Chemicals		Proprietary Chemicals		
	Calcium Polysulfide	Sodium Sulfide	Ferrous Sulfide (FerroBlack®)	Ferrous Sulfide (FerroBlack®) and Calcium Oxide	Molecular Bonding System (MBS®)
Relative Cost/kg of Hg* (assuming Hg 6000mg/kg soil)	<ul style="list-style-type: none">• minimum \$0.77• maximum \$5.13	<ul style="list-style-type: none">• minimum \$1.38• maximum \$36.67	<ul style="list-style-type: none">• minimum \$2.86• maximum \$21.45	<ul style="list-style-type: none">• minimum \$5.06• maximum \$23.65	<ul style="list-style-type: none">• minimum \$5.50• maximum \$18.33
Handling and Delivery Considerations	Difficult <ul style="list-style-type: none">• Strong odors• Caustic	Difficult <ul style="list-style-type: none">• Caustic• Challenges with mixing and slurry generation• High pH must be maintained	Medium <ul style="list-style-type: none">• Addition of FerroBlack, which is solid particles suspended in liquid	Medium <ul style="list-style-type: none">• Recommended application of Calcium oxide first, monitor until a temperature of 90 is reached• Followed by addition of FerroBlack, which is solid particles suspended in liquid	Easy <ul style="list-style-type: none">• Mixing of non-hazardous solid material• Can add water to aid in mixing

* Range in quantities based on range of concentrations of amendments to be tested in bench scale treatability study workplan (and suggestions by vendors)

¹ Price quote provided by commercial supplier listed

² Price quoted from internet search

Appendix B

SDL Modeling and Evaluations

Appendix B

SDL Modeling and Evaluations

To better represent actual leaching in the field, Semi-dynamic Leaching (SDL) was performed at the DTL using a modified SW-846 method 1315 and ASTM method 1308 (SOP 1-4). Each solidified/stabilized sample was immersed in synthetic rainwater (SPLP water) in a polytetrafluoroethylene (i.e., Teflon) container. The solidified/stabilized sample surface area to water ratio was 1:9 (centimeters to milliliter). The leachate was removed from the vessel and replaced with fresh SPLP water at the following time intervals: 2 hours, 24 hours, 48 hours, 72 hours, 8 days, 14 days, 21 days, 28 days and 42 days. Table 6-8 in the main text provides the solidified/stabilized sample treatments selected for SDL testing and includes the dimensions and surface areas of each core leached. Table 6-9 in the main text provides the mass of water used to create the leachate at each exchange interval as well as measurement parameters including pH, ORP, and conductivity performed at the DTL during this procedure. Table 6-10 in the main text provides results of the dissolved mercury analysis performed by Chemtech Consulting Group. These results are also attached to this Appendix as **Table B-1**.

As shown in **Table B-1**, dissolved mercury results for SS-H-CPx-10-Solidified ranged from 1.2 µg/L at 21 days to 209 µg/L at 24 hours. For sample SS-H-FB-15-Solidified, dissolved mercury ranged from 6.4 µg/L at 28 days to 231 µg/L at 2 hours. For sample SS-H-CaO-FB-10-Solidified, dissolved mercury ranged from 12 µg/L at 28 days to 329 µg/L at 2 hours.

Overall the dissolved mercury results from SS-H-CPx-10-Solidified sample were very low during the last five leaching times (8 to 42 days) ranging from 0.88 to 2.9 µg/L. Evaluation of the SDL results (see below) indicate that the initial higher concentrations are the result of surface wash off while the later lower concentrations are the result of depletion and/or dissolution of the mercury from the stabilized/solidified samples. Based on the SDL tests, the estimated mercury concentrations at the interface of water (e.g., groundwater) and the stabilized/solidified soil would be approximately 3 to 4 µg/L over the time period of 2 to 10 years. These concentrations would decrease away from the water/soil interface as a result of additional mixing with the groundwater.

The objective of the SW-846 method 1315 leaching procedure is to provide “material parameters” (e.g., concentrations, mass quantities, release rates, etc. from the solidified/stabilized sample) for the release of the contaminant of concern (mercury) from the material (solidified/stabilized sample) to the eluent (leaching solution) under controlled leaching conditions. Evaluations of the Method 1315 results and total sample concentrations are used to determine the release mechanisms of the mercury from the solidified/stabilized samples. These mechanisms can include surface wash off, diffusion, depletion of mass, dissolution, or a combination of these mechanisms over different time. The evaluations are based on a plot of the log of the cumulative mass released vs the log of the leaching time. When the mechanism(s) is identified and the release rates are “well behaved” (i.e., the equations from the plots have a reasonable coefficient of determination, r^2), contaminant aqueous phase concentrations into the future (years) at the water/exposed surface of the solidified/stabilized material interface can be predicted.

When diffusion is the controlling mechanism, a plot of the logarithm of the cumulative mass released vs the logarithm of time should have a slope (rc) of 0.5 ± 0.15 (>0.35 to <0.65). In the current evaluation, the

slope between each successive interval (called the “EPA slope”) was calculated using the interval in question (i) and the previous interval (i-1). Intervals which deviate from the ideal diffusion slope are characterized by another release process. For example, the first leaching intervals may be influenced by “surface wash-off”. Contaminants of concern (mercury) which are at the surface of the sample can be released by desorption or dissolution alone without diffusion through the sample matrix. Intervals near the end of the test may be affected by “depletion”, in which the near surface contaminant (mercury) concentrations become depleted. The European Union “TANK” procedure (EA, 2005) provides a detailed procedure to identify the relevant release processes for specific sets of intervals. The processes were evaluated using the “Tank” methodology by calculating the slope (rc) between specific time intervals (see **Table B-2**) of the plot of the log of the cumulative mass released vs the log of the leaching time. Release mechanisms were identified for the various time intervals based on the calculated slope (see **Table B-2**).

To complete the evaluations, several calculations were required and summarized in the following paragraphs.

The mass of contaminant of concern released for each interval (M_{ti}) was calculated as follows:

$$M_{ti} = C_i V_i / A$$

Where,

C_i = The concentration of the contaminant within the leachate solution for interval i (mg/L)

V_i = The volume of leaching solution used for interval i (L)

A = The surface area of the sample (m^2)

These data were used to calculate the sample volumes using the following:

$$V_{\text{sample}} = h\pi r^2$$

where

V_{sample} = The volume of the sample in m^3

h = The height of the sample in m

r = The radius of the sample in m

The dry bulk density (ρ) was also calculated (see Table 6-13 in main report) using the volumes calculated above, the core masses and the measured moisture contents using the following:

$$\rho = (M_{\text{soil-wet}} * (1 - (\phi/100\%))) / V_{\text{sample}}$$

where,

$M_{\text{soil-wet}}$ = The wet mass of the soil (kg)

ϕ = The percent moisture of the soil (water mass/wet soil mass * 100%)

The water content as used here was the variable defined in the geological sciences, which is the mass of water divided by the total mass times 100%. The water content used in soil mechanics is the mass of water divided by the dry mass of soil (see Table 6-13 in main text).

The surface area of the samples was calculated as follows (see Table 6-8 in main report):

$$A = 2\pi rh + 2\pi r^2$$

The resulting calculations and conclusions for SS-H-CPx-10-Solidified are provided in the **Table B-3**. As shown, after initial relatively high concentrations due to wash off, the lower concentrations are controlled by dissolution or depletion. For evaluating future concentrations, the first two intervals were not used due to initial wash off resulting in a plot with coefficient of determination (r^2) of 0.84 (see graph on **Table B-3**). This equation was used to predict future concentrations between 1 and 10 years at the interface between the solidified/stabilized soil and the aqueous phase (groundwater). The concentration at one year was 9.2 ug/L. The concentrations from years 2 to 10 ranged from 3.3 to 4.4 ug/L dissolved mercury. These concentrations would decrease as the water at the interface migrates and mixes with additional groundwater.

The above analyses were also performed for the SDL results from samples SS-H-FB-15-Solidified and SS-H-CaO-FB-10-Solidified. The associated graphs and release mechanisms are provided in **Table B-4**. These results are not discussed in detail because FerroBlack was not selected for any pilot scale testing due to its high cost, difficulty in implementation and relatively higher initial and ending mercury concentrations.

References

ASTM. 2008. Method C1308-08, *Standard Test Method for Accelerated Test for Diffusive Releases from Solidified Waste with a Computer Program to Model Diffusive, Fractional Leaching from Cylindrical Waste Forms*, ASTM International, West Conshohocken, PA, DOI: 10.1520/C1308-08.

EA, 2005. *Leaching Characteristics of Moulded or Monolithic Building and Waste Materials. Determination of Leaching of Inorganic Components with the Diffusion Test. 'The Tank Test'*. Environmental Agency EA NEN 7375:2004. April, 2005.

EPA, 2013. SW-846 Method 1315 – *Mass Transfer Rates of Constituents in Monolithic or compacted Granular Materials Using a Semi-Dynamic Tank Leaching Procedure*. January 2013.

Table B-1 Semi-Dynamic Leaching Results

Sample ID	Time Interval	Mercury (µg/L)	
		Result	Q
SS-H-CPx-10-Solidified	2 hours	16	
	24 hours	209	
	48 hours	70	
	72 hours	8.9	
	8 days	1.3	
	14 days	0.88	
	21 days	1.2	
	28 days	2.2	
	42 days	2.9	
SS-H-FB-15-Solidified	2 hours	231	
	24 hours	5.4	
	48 hours	2.5	
	72 hours	2.7	
	8 days	3.7	
	14 days	8	
	21 days	7.6	
	28 days	6.4	
	42 days	44	
SS-H-CaO-FB-10-Solidified	2 hours	329	
	24 hours	182	
	48 hours	112	
	72 hours	57	
	8 days	24	
	14 days	19	
	21 days	46	
	28 days	12	
	42 days	124	

Notes:

µg/L = micrograms per liter

Q = qualifier

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation

Table B-2 Release Mechanisms (Table 3 in EA 2005)

Interval a to b	Slope (rc)		
	< 0.35	>0.35 to <0.65	>0.65
Interval 2-7	Surface wash off	Diffusion	Dissolution
Interval 5-8	Depletion	Diffusion	Dissolution
Interval 4-7	Depletion	Diffusion	Dissolution
Interval 3-6	Depletion	Diffusion	Dissolution
Interval 2-5	Depletion	Diffusion	Dissolution
Interval 1-4	Surface wash-off	Diffusion	Delayed diffusion or dissolution

Table B-3 Evaluation of SDL Mercury Concentrations for SS-H-CPx-10-Solidified

Interval	t (sec) (interval)	t (sec) (cumulative)	Days	Mti (E*i)	Mt-cumlt (ε*n)	Solution Mass (g)	Log[Mt]	Log t	C(soln) mg/L	C(soln) ug/L	EPA slope	εn (mg/m2)	log εn (mg/m2)
1	7200	7200	0.083333	1.343	1.343	869.7	0.128	3.86	0.01590	15.9		1.343	0.128
2	79200	86400	1	17.424	18.767	858.7	1.273	4.94	0.20900	209.0	1.061	24.495	1.389
3	86400	172800	2	5.848	24.615	865.5	1.391	5.24	0.06960	69.6	0.391	19.968	1.300
4	86400	259200	3	0.758	25.373	877.5	1.404	5.41	0.00890	8.9	0.075	4.132	0.616
5	432000	691200	8	0.112	25.486	890.4	1.406	5.84	0.00130	1.3	0.005	0.290	-0.538
6	518400	1209600	14	0.076	25.561	886.8	1.408	6.08	0.00088	0.9	0.005	0.310	-0.508
7	604800	1814400	21	0.105	25.667	904.3	1.409	6.26	0.00120	1.2	0.010	0.574	-0.241
8	604800	2419200	28	0.197	25.864	921.6	1.413	6.38	0.00220	2.2	0.027	1.469	0.167
9	1209600	3628800	42	0.260	26.123	922.6	1.417	6.56	0.00290	2.9	0.025	1.416	0.000

EU Tank Leaching Mechanisms				
Interval	Average Interval Conc	Interval Slope(rc)	Std Dev rc	Conclusion
2 - 7	96.96	-1.582	0.077	Surface Wash Off
5 - 8	2.79	1.222	0.076	Dissolution
4 - 7	6.14	-1.112	0.063	Depletion
3 - 6	40.34	-1.956	0.076	Depletion
2 - 5	144.40	-2.256	0.107	Depletion

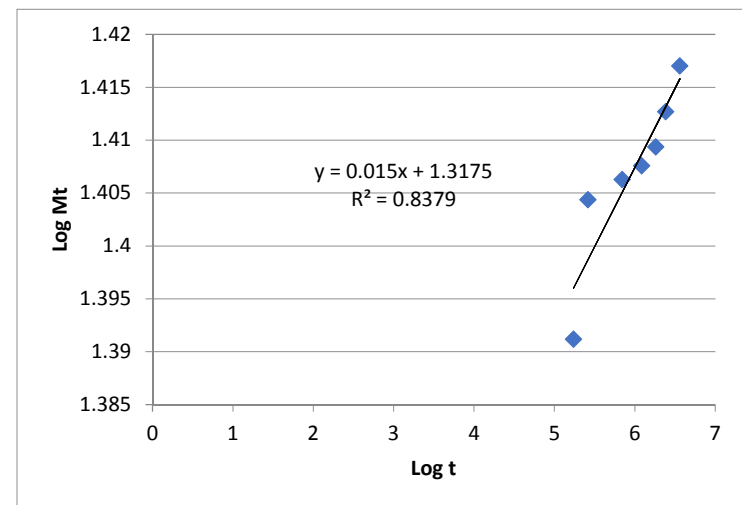
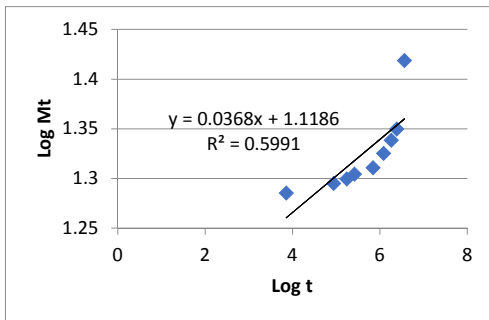


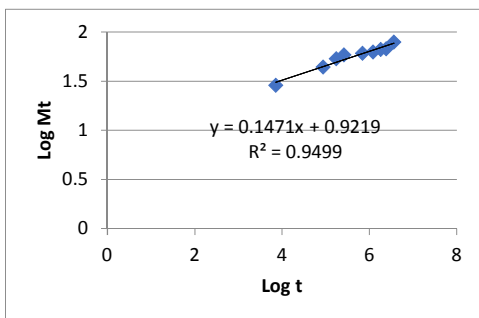
Table B-4 SDL Evaluations

SS-H-FB-15-Solidified



EU Tank Leaching Mechanisms				
Increment	Average Conc	Slope(rc)	SDrc	Conclusion
2 - 7	9.97	0.552	0.077	Diffusion
5 - 8	12.85	1.303	0.076	Dissolution
4 - 7	11.00	0.628	0.063	Diffusion
3 - 6	8.45	0.454	0.076	Diffusion
2 - 5	7.15	0.164	0.107	Depletion
1 - 4	120.80	-0.906	0.100	Surface Wash Off

SS-H-CaO-FB-10-Solidified



EU Tank Leaching Mechanisms				
Increment	Average Conc	Slope(rc)	SDrc	Conclusion
2 - 7	146.57	-0.323	0.077	Surface Wash Off
5 - 8	50.10	0.594	0.076	Diffusion
4 - 7	72.85	-0.239	0.063	Depletion
3 - 6	106.05	-0.636	0.076	Depletion
2 - 5	187.80	-0.691	0.107	Depletion
1 - 4	340.10	-0.002	0.100	Surface Wash Off

Appendix C

Subcontract Laboratory (Brooks Applied Labs) Report

**CDM FEDERAL PROGRAMS CORP.
LCP CHEMICAL INC
SUPERFUND SITE RD
SK5807**

**KATAHDIN ANALYTICAL SERVICES
600 TECHNOLOGY WAY
SCARBOROUGH, ME 04074**



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SUBCONTRACTED DATA

Paginated by
Sub Lab

SAMPLE DATA PACKAGE

NARRATIVE
KATAHDIN ANALYTICAL SERVICES
CDM FEDERAL PROGRAMS CORPORATION
LCP CHEMICAL INC SUPERFUND SITE RD
SK5807

Sample Receipt

The following samples were received on July 05, 2017 and were logged in under Katahdin Analytical Services work order number SK5807 for a hardcopy due date of July 23, 2017.

KATAHDIN <u>Sample No.</u>	CDM <u>Sample Identification</u>
SK5807-1	SS-H-6000-051917
SK5807-2	SS-H-96000-051917
SK5807-3	SS-M-1500-051717
SK5807-4	SS-H-CPX-10
SK5807-5	SS-H-CPX-10-OPTI
SK5807-6	SS-H-CPX-10-SOLIDIFIED
SK5807-7	SS-H-FB-15-OPTI
SK5807-8	SS-H-FB-15-SOLIDIFIED
SK5807-9	SS-H-FB-CAO-10-SOLIDIFIED
SK5807-10	SS-H-S-25
SK5807-11	SS-H-S-5
SK5807-12	SS-M-CPX-5
SK5807-13	SS-M-CPX-5-OPTI
SK5807-14	SS-M-FB-5
SK5807-15	SS-M-FB-CAO-5
SK5807-16	SS-M-FB-CAO-5-OPTI

The samples were logged in for the analyses specified on the chain of custody form. All problems encountered and resolved during sample receipt have been documented on the applicable chain of custody forms.

We certify that the test results provided in this report meet all the requirements of the NELAC standards unless otherwise noted in this narrative or in the Report of Analysis.

Sample analyses have been performed by the methods as noted herein.

Should you have any questions or comments concerning this Report of Analysis, please do not hesitate to contact your Katahdin Analytical Services Project Manager, **Ms. Heather Manz**. This narrative is an integral part of the Report of Analysis.

Subcontracted Data

Analyses for Total Mercury by Method 1631, Total Solids by Method SM2540G, and Mercury Speciation were performed by a subcontract laboratory. Please refer to the section of the data package titled Subcontracted Data.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Quality Assurance Officer, or their designee, as verified by the following signature.

Heather Manz for H. Manz 08/09/17

Heather Manz
Project Manager

CHAIN OF CUSTODY RECORD

No: 2-052317-181528-0039

Lab: Brooks Applied Labs

Lab Contact: Tiffany Stilwater

Lab Phone: 206-632-6206

Cooler #:

Sample(s) to be used for Lab QC: SS-M-1500-051717 Tag A	Shipment for Case Complete? N
Analysis Key: THg/HgSp=Total Hg/Hg Speciation	Samples Transferred From Chain of Custody #

[illegible]

0000006

USEPA CLP COC (LAB COPY)

Date Shipped: 6/29/2017

Carrier Name: FedEx

Airbill No: 779516647548

CHAIN OF CUSTODY RECORD

No: 2-062817-131507-0046

Lab: Brooks Applied Labs

Lab Contact: Tiffany Stillwater

Lab Phone: 206-632-6206

DAS #:

Cooler #:

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	For Lab Use Only
SS-H-CPX-10		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 12:45	
SS-H-CPX-10-OPTI		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 13:00	
SS-H-CPX-10-Solidified		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 11:00	
SS-H-FB-15-OPTI		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 13:20	
SS-H-FB-15-Solidified		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 11:45	
SS-H-FB-CaO-10-Solidified		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 12:14	
SS-H-S-25		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 12:30	
SS-H-S-5		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 12:55	
SS-M-CPX-5		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 12:50	
SS-M-CPX-5-OPTI		SO/ T. Burgesser	Grab	THg/HgSp(21)	A (4 C) (1)	Bench Scale Area	06/09/2017 13:10	

Shipment for Case Complete? N

Samples Transferred From Chain of Custody #

Sample(s) to be used for Lab QC: SS-H-CPX-10-Solidified Tag A - Special Instructions: Matrix Code: SO = Soil

Analysis Key: THg/HgSp=Total Hg/Hg Speciation

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	<i>See below</i> / COC-Suite L	6/29/17 1500	<i>[Signature]</i>	6/30/17 0930	

0000007

USEPA CLP COC (LAB COPY)

DateShipped: 6/29/2017
CarrierName: FedEx
AirbillNo: 779516647548

CHAIN OF CUSTODY RECORD

No: 2-062817-131507-0046

Lab: Brooks Applied Labs
Lab Contact: Tiffany Stilwater
Lab Phone: 206-632-6206

[illegible]

	Shipment for Case Complete? N
	Samples Transferred From Chain of Custody #
Special Instructions: Matrix Code; SO = Soil	
Analysis Key: THg/HgSp=Total Hg/Hg Speciation	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	<i>James L. Smith</i>	06/29/17 1530	<i>[Signature]</i>	6/30/17 0930	

oooooooo

Katahdin Analytical Services

Login Chain of Custody Report (Ino1)

Page: 1 of 2

Jul. 05, 2017

04:53 PM

Login Number: SK5807

Quote/Incoming:

Account: CDMFED003

NoWeb

CDM Federal Programs Corporation

Project:

Primary Report Address:

 Muzaffar (Ali) Rahmani
 CDM Smith
 Raritan Plaza I
 110 Fieldcrest Ave, #8, 6th floor
 Edison, NJ 08837

rahmanima@cdmsmith.com

Primary Invoice Address:

 Accounts Payable
 CDM Federal Programs Corporation
 3201 Jermantown Road
 Suite 400
 Fairfax, VA 22030

Report CC Addresses:
Invoice CC Addresses:
Login Information:

ANALYSIS INSTRUCTIONS : Mercury-sub: Mercury Speciation. SW7470 Sub: E1631-Trace Mercury. All direct subbed to Brooks Applied.

 CHECK NO. :
 CLIENT PO# : 1110-000-014-AL/Task Order 08/20721

CLIENT PROJECT MANAGE :

CONTRACT :

COOLER TEMPERATURE : sub only

DELIVERY SERVICES : sub only

EDD FORMAT : KAS138QC-CSV

LOGIN INITIALS : AMH

PM : HHM

PROJECT NAME : LCP Chemical Inc Superfund Site RD

QC LEVEL : IV

REPORT INSTRUCTIONS : Send final PDF and EDD to Graham Reifert (ReifertGB@cdmsmith.com),

SDG ID :

SDG STATUS :

VERBAL TAT :

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Mailed
SK5807-1	SS-H-6000-051917	19-MAY-17 14:00	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	16-JUN-17	500mL Plastic+HNO3				
SK5807-2	SS-H-96000-051917	19-MAY-17 14:00	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	16-JUN-17	500mL Plastic+HNO3				
SK5807-3	SS-M-1500-051717	17-MAY-17 15:00	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	14-JUN-17	500mL Plastic+HNO3				
SK5807-4	SS-H-CPX-10	09-JUN-17 12:45	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3				
SK5807-5	SS-H-CPX-10-OPTI	09-JUN-17 13:00	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3				
SK5807-6	SS-H-CPX-10-SOLIDIFIED	09-JUN-17 11:00	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3				
SK5807-7	SS-H-FB-15-OPTI	09-JUN-17 13:20	05-JUL-17			23-JUL-17	
Matrix	Product	Hold Date (shortest)	Bottle Type	Bottle Count	Comments		
Aqueous	S MERCURY-SUB						
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3				

0000009

Login Number: SK5807

Account: CDMFED003

NoWeb

CDM Federal Programs Corporation

Project:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	Verbal PR Date	Due Date	Mailed
SK5807-8	SS-H-FB-15-SOLIDIFIED	09-JUN-17 11:45	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-9	SS-H-FB-CAO-10-SOLIDIFIED	09-JUN-17 12:14	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-10	SS-H-S-25	09-JUN-17 12:30	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-11	SS-H-S-5	09-JUN-17 12:55	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-12	SS-M-CPX-5	09-JUN-17 12:50	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-13	SS-M-CPX-5-OPTI	09-JUN-17 13:10	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-14	SS-M-FB-5	09-JUN-17 12:40	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-15	SS-M-FB-CAO-5	09-JUN-17 12:45	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			
SK5807-16	SS-M-FB-CAO-5-OPTI	09-JUN-17 13:30	05-JUL-17		23-JUL-17	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>	<i>Comments</i>	
Aqueous	S MERCURY-SUB					
Aqueous	S SW7470-MERCURY-SUB	07-JUL-17	500mL Plastic+HNO3			

Total Samples: 16

Total Analyses: 32

0000010

SUBCONTRACTED DATA

Report of Mercury Speciation Analyses Soil

Project: LCP Chemical Superfund Site

Samples Collected: May 19 – June 9th, 2017

Report Date: August 3, 2017

Prepared for:

Allison Harbottle

Katahdin Analytical Services

600 Technology Way

Scarborough, ME 04074

Project ID: KAT-SC1701

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Case Narrative

Shipping and Receiving

On May 25, 2017, Brooks Applied Labs (BAL) received three (3) soil samples at 8:25 A.M. in a cooler with blue ice at a temperature of 1.0°C. Thirteen more soil samples were received on June 30, 2017 at 9:30 A.M. in a cooler with ice at a temperature of 2.9°C. The chain-of-custody (COC) forms indicated analysis for total mercury (Hg), methyl mercury (MeHg), percent total solids (%TS), and mercury by five-step selective sequential extraction (SSE).

The samples were received and stored securely according to Brooks Applied Labs (BAL) standard operating procedures (SOP) and EPA methodology.

Preservation and Holding Time

All method and SOP requirements for preservation and holding time were satisfied.

Total Mercury in Sediment/Soil by EPA Method 1631 (SOP BAL-3101)

All samples are prepared and analyzed in accordance with EPA Method 1631. Samples are digested with aqua regia at room temperature, oxidized with bromine monochloride (BrCl), and then analyzed with stannous chloride (SnCl₂) reduction, single gold amalgamation, and cold vapor atomic fluorescence spectroscopy (CVAFS) detection using a Brooks Rand Instruments MERX-T CVAFS Mercury Automated-Analyzer.

The results were method blank-corrected as described in the calculations section of the relevant BAL SOP(s) and may have been evaluated using reporting limits that have been adjusted to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific MDLs, MRLs, and other details.

Sequence 1700837

Continuing calibration blank CCBA, CCBC, and CCBF recover high. All samples for total mercury had results more than ten times the high bracketing CCBs and therefore are not affected. Further instrument calibration, meeting all quality control criteria, was successfully achieved on the day of sample analysis.

Batch B171654

The matrix spikes and matrix spike duplicates (B171654-MS2/MSD2 and B171654MS3/MSD3) associated with this batch were under spiked and therefore are not valid indicators of data quality. No corrective action is necessary. All data was reported without qualification and all other associated quality control sample results met the acceptance criteria.

Percent Total Solids in Solids by SM 2540G (SOP BAL-0501)

A known mass of each soil sample was placed into a pre-weighed pan, then the combined mass of the sample and pan was recorded. All samples were placed into a convection oven maintained at a temperature of 105°C. After drying for a minimum of 48 hours all samples were briefly cooled and reweighed. The total solids percentage of each sample was calculated by dividing the weight of the dried sample by the weight of the original sample.

Batch B171661

All data was reported without qualification and all associated quality control sample results met the acceptance criteria.

Mercury Speciation in Soils by 5-Step Extraction (SOP BAL-3900)

Mercury is extracted from an accurately weighed sediment sample into five different solutions that can be broadly linked to types of mercury compounds. The extractants used are: deionized water (F1), a synthetic “stomach acid” (F2), 1M potassium hydroxide solution (F3), 12M nitric acid (F4), and aqua regia (F5). All samples are analyzed in accordance with EPA Method 1631. Samples are oxidized with bromine monochloride (BrCl) and then analyzed with stannous chloride (SnCl_2) reduction, single gold amalgamation, and cold vapor atomic fluorescence spectroscopy (CVAFS) detection using a Brooks Rand Instruments MERX-T CVAFS Mercury Automated-Analyzer.

The results were method blank-corrected as described in the calculations section of the relevant BAL SOP(s) and may have been evaluated using reporting limits that have been adjusted to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific MDLs, MRLs, and other details.

General Method Comments

Experience with this method has demonstrated that quantified results for both samples and SRMs can be somewhat variable and therefore the method is not completely effective for determining the concentrations of individual fractions in each sample. However, this method has been shown to be effective as a qualitative assessment of the relative percentages of each mercury fraction. Given this information, data quality evaluation should focus on the analysis of the percentage of total Hg rather than concentrations or recoveries within any particular fraction.

No certified reference materials for the SSE Procedure are commercially available. The reference materials Hg^0 , HgS , and HgCl_2 were produced by Studio Geochemica (SGC) and are not officially certified for any analyte. SGC provided expected concentrations for each fraction of the SSE procedure as well as total Hg concentrations and these values have largely been confirmed by analysis at BAL. As such, BAL utilizes these custom reference materials with expected values of mercury to be found at each step of the process. The results from each fraction are then summed and compared against their expected total mercury concentrations. The standard reference materials (SRM) analyzed at each step of the extraction produced recoveries outside of the typical solid matrix control limits for total mercury (THg) analysis. As no control limits have been officially established for the recoveries of SRMs for the SSE Procedure, the recoveries were not indicative of poor data quality. The recovered percent of each SRM should be compared to the expected percent of SRM (as determined by historical results obtained by BAL and/or Studio Geochemica). Furthermore, the sum of all Hg fractions should be compared against the certified total Hg value to demonstrate the efficiency of the SSE Procedure at recovering all available forms of Hg.

Table 1 outlines the expected SRM percent recovery compared to the achieved SRM percent recovery for each fraction of the SSE Procedure. Over time, BAL has observed a shift in some of the recoveries due to SRM degradation. Notably, the recovery of Hg from HgCl_2 SRM has shifted from the F1 step to the F2 and F3 steps. Additionally, more of the Hg in the HgS SRM is now available in the F3 step, making less available in the F5 step. NIST2710 mainly recovers in the fifth step historically, which aligns with recoveries in this preparation. HgS recovers mainly in F5, although it recovers higher than expected in the F3 fraction. Hg^0 recovers mainly in F4, although higher than expected in F1 and F3, accounting for the lower recovery in F4. Historically, HgCl_2 recovers mainly in F1. In this instance the F3 recovery is higher than expected. The recoveries highlighted in gold are recoveries that differ from expected recoveries.

SRM	F1		F2		F3		F4		F5	
Name	exp.%	rec. %	exp.%	rec. %	exp.%	rec. %	exp.%	rec. %	exp.%	rec. %
NIST 2710	0.6%	0.6%	0.1%	0.3%	2.2%	2.3%	43.0%	72.8%	50.4%	39.6%
HgS	0.0%	0.0%	0.0%	0.0%	0.0%	4.2%	0.4%	1.8%	93.8%	80.2%
Hg0	1.3%	13.2%	1.3%	1.7%	6.7%	10.0%	89.4%	72.1%	0.3%	0.5%
HgCl2	75.5%	40.9%	11.4%	10.3%	4.2%	15.0%	6.3%	10.0%	1.1%	1.6%

Table 1: Table comparing the expected % of total (exp. %) to the recovered percent of total (rec. %) for the SRMs prepared with the 5-step sequential extraction.

Table 2 outlines the sums of the fractions for each CRM were compared to the established expected values in the table below. The aggregation of the fractions produced a recovery within the acceptance criteria for all reference materials.

SRM Name	Sum of Results	Sum of expected results	Recovery
NIST 2710	36542	31600	116%
HgS	1744329	2023000	86%
Hg0	5661910	5804000	98%
HgCl2	1454750	1872000	78%

Table 2: The % recovery (criteria: recovery = 75% - 125%) based on the sum of the analyzed results for each of the 5 fractions and the certified total Hg value for each reference material. Results are in ng/g.

Table 3 compares the sum of the SSE fractions to the THg results for each sample. It is important to note that the 5-step procedure is not designed to provide total numbers for Hg in the sample as much as it is designed to show how extractable the Hg is from the sample. As such, it is not uncommon for the total numbers to not perfectly match the sum of the species.

Poor RPDs were observed for the samples highlighted below. In the case of 1721026-03, and 1721036-16 the sum of the 5 step extraction results exceeds the total Hg result. In the case of 1721026-08, 1721026-10, 1721026-13, and 1721026-14, the sum of 5 step extraction results is less than the total Hg result.

Sample ID	F1	F2	F3	F4	F5	Total Hg Result*	SUM of 5 step	RPD
1721026-01	23700	280000	405000	2940000	2610000	6990000	6258700	11%
1721026-02	30900	335000	493000	3820000	3130000	6910000	7808900	12%
1721026-03	12900	5660	20300	1080000	2940000	2300000	4058860	55%
1721026-04	44400	121	44800	423000	5970000	5990000	6482321	8%
1721026-05	304000	368	32100	3040000	2890000	7610000	6266468	19%
1721026-06	30.6	23200	9610	2820000	2390000	5340000	5242841	2%
1721026-07	1110	871	21800	2770000	1140000	4840000	3933781	21%
1721026-08	420000	461	181000	2770000	2670000	8830000	6041461	38%
1721026-09	354000	39200	6480	949000	1870000	4120000	3218680	25%
1721026-10	8870	49400	86500	3230000	1970000	7830000	5344770	38%
1721026-11	11400	79100	9500	3920000	2320000	6280000	6340000	1%
1721026-12	10400	176	27000	363000	1630000	1840000	2030576	10%
1721026-13	742	255	12300	599000	842000	2830000	1454297	64%
1721026-14	4260	259	6380	528000	1200000	2740000	1738899	45%
1721026-15	4550	310	5290	209000	1520000	1790000	1739150	3%
1721026-16	557	120	5580	519000	89900	4070000	615157	147%

Table 3: Comparing the sum of the 5-step fraction results to the total Hg result. Results are in ng/g. (Criteria: RPD ≤ 35%)

	F1	F2	F3	F4	F5
Sample ID	% of sum	% of sum	% of sum	% of sum	% of sum
1721026-01	0.4%	4.5%	6.5%	47.0%	41.7%
1721026-02	0.4%	4.3%	6.3%	48.9%	40.1%
1721026-03	0.3%	0.1%	0.5%	26.6%	72.4%
1721026-04	0.7%	0.0%	0.7%	6.5%	92.1%
1721026-05	4.9%	0.0%	0.5%	48.5%	46.1%
1721026-06	0.0%	0.4%	0.2%	53.8%	45.6%
1721026-07	0.0%	0.0%	0.6%	70.4%	29.0%
1721026-08	7.0%	0.0%	3.0%	45.8%	44.2%
1721026-09	11.0%	1.2%	0.2%	29.5%	58.1%
1721026-10	0.2%	0.9%	1.6%	60.4%	36.9%
1721026-11	0.2%	1.2%	0.1%	61.8%	36.6%
1721026-12	0.5%	0.0%	1.3%	17.9%	80.3%
1721026-13	0.1%	0.0%	0.8%	41.2%	57.9%
1721026-14	0.2%	0.0%	0.4%	30.4%	69.0%
1721026-15	0.3%	0.0%	0.3%	12.0%	87.4%
1721026-16	0.1%	0.0%	0.9%	84.4%	14.6%

Table 4. Fraction percentages of the samples.

Please refer to Table 4 to indicate the mercury fraction percentages for each sample. For all samples, the mercury was primarily available in the F4 and F5 extractions. The fraction where Hg was mainly extracted is highlighted green; the next largest extracted fraction is highlighted blue.

Sequence 1700825 (F0)

Samples 1721026-05, -08, -09, -12 yielded results over calibration and have been qualified **J-1**. Samples 1721026-01, -02, -06, -10, -13 and -14 were analyzed after the high calibration samples and may be affected by carryover; however, no qualification was applied.

Continuing calibration blanks CCBD and CCBE yielded results above the calibration standard. Samples 1721026-06, -07, -13, -14, and -16 were bracketed by these CCBs and had results less than 10 times the CCBs and may have been affected. The F0 step of the extraction is not able to be reanalyzed due to the nature of the analysis. As such, since F0 is a qualitative test samples have not been qualified. Instrument calibration, meeting all other quality control criteria, was successfully achieved on the day of sample analysis.

Batch B171685 (F0)

The first duplicate (DUP1) was outside of RPD criteria, and secondary criteria was not met. The F0 step of extraction is not able to be reanalyzed, and as a result source sample *SS-M-1500-051717* (1721026-03) has been qualified **M**. All other data was reported without qualification and all other associated quality control sample results meet the acceptance criteria.

Sequence 1700837 (F1, F2, F3)

Continuing calibration blanks CCBA, CCBC, and CCBF recover high. Samples bracketed by the high blanks with results above the MRL and less than ten times the hit the blanks were reanalyzed. Additionally, several samples were over calibration. These samples, including the samples subsequent to the over calibration samples were reanalyzed. Instrument calibration, meeting all other quality control criteria, was successfully achieved on the day of sample analysis.

Sequence 1700846 (F4, F5)

The third calibration standard (CAL3) was spiked at 250pg instead of 100pg. The BAL Quality Assurance Officer approved the deviation. Instrument calibration, meeting all quality control criteria, was successfully achieved on the day of sample analysis.

Sequence 1700862 (F1, F2, F3, F4, F5)

Continuing calibration blank CCB5 recovers high. Sample 1721026-07 was bracketed by the high blank with a F1 result above the MRL and less than ten times the hit the blanks was reanalyzed. Instrument calibration, meeting all other quality control criteria, was successfully achieved on the day of sample analysis.

Sequence 1700870 (F2)

Continuing calibration blank (CCB) CCB9 yielded a result above the calibration standard. No samples from this work order were bracketed by this CCB. Instrument calibration, meeting all other quality control criteria, was successfully achieved on the day of sample analysis.

Sequence 1700875 (F1)

The first calibration standard was possibly double spiked. This sample was re-prepared and reanalyzed as CAL 7 and CAL8. CAL 7 has been reported. Instrument calibration, meeting all quality control criteria, was successfully achieved on the day of sample analysis.

Sequence 1700882 (F1)

Instrument calibration, meeting all quality control criteria, was successfully achieved on the day of sample analysis.

Batch B171656 (F1)

Method blank B171656-BLK1 recovered at 1.43 ng/g and was determined to be a Grubb's outlier at less than 5% risk of false rejection. All samples were evaluated against the Grubb's outlier, and all detect samples recovered above ten times the level of the Grubb's, therefore no qualification is necessary. The first duplicate (DUP1) and post spike (PS1) recovered outside of RPD criteria and did not meet secondary criteria. The source sample *SS-M-1500-051717* (1721026-03) and its associated quality control samples were reanalyzed. The re-analysis confirmed the initial duplicate RPD (DUP3), however the post spike (PS3) recovery performed well. The results from the re-analysis have been reported, and the source sample (1721026-03) has been qualified **M** for duplicate imprecision. Sample *SS-H-CPx-10-Solidified* (1721026-06) and its corresponding QC were re-analyzed due to potential carryover/bleed-back from high bracketing CCBs. Re-analyses confirmed sample results, however the associated PS recovered low. Sample results have been qualified **N** and should be considered an estimate. All other data was reported without further qualification and all other associated quality control sample results meet the acceptance criteria.

Batch B171657 (F2)

Method blank B171657-BLK1 recovered at 55.3 ng/g and was determined to be a Grubb's outlier at less than 0.1% risk of false rejection. All detect samples recovered above ten times the level of the Grubb's, therefore no qualification is necessary. The second duplicate (DUP2) recovered outside of RPD criteria and did not meet secondary criteria. The source sample *SS-H-CPx-10-Solidified* (1721026-06) and its associated quality control were reanalyzed for confirmation. Re-analysis as DUP4 confirmed the initial results and has been reported. The source sample (1721026-06) has been qualified **M** for duplicate imprecision. The first post spike (PS1) also recovered outside of criteria. The source sample *SS-M-1500-051717* (1721026-03) and corresponding QC were re-analyzed. The re-analyses of the PS yielded good recoveries, however the DUP RPD did not confirm the initial results and produced a poor RPD. A third re-analysis was performed which confirmed the DUP RPD. The fifth duplicate (DUP5) and post spike (PS5) have been reported and the source sample (1721026-03) has been qualified **M** for duplicate imprecision.

Batch B171658 (F3)

Method blank BLK4 recovered at 16.7 ng/g and was determined to be a Grubb's outlier at less than 5% risk of false rejection. All samples all detect samples recovered above ten times the level of the Grubb's, therefore no qualification is necessary. The first duplicate (DUP1) and post spike (PS1) recovered outside of RPD criteria and did not meet secondary criteria. The source sample *SS-M-1500-051717* (1721026-03) and its associated quality control were reanalyzed for confirmation and confirmed failing RPD. The third DUP (DUP3) has been reported and the source sample qualified **M**. The second duplicate (DUP2) also recovered outside of RPD criteria and secondary criteria was not met. Re-analyses of the source sample *SS-H-CPx-10-Solidified*

(1721026-06) confirmed failing RPD. The fourth duplicate (DUP4) has been reported, and the source sample qualified **M**. The third and fourth post spikes (PS3/PS4) were spiked below the source sample concentrations. Therefore, PS recoveries are not valid indicators of data quality. No qualification is required. All data was reported without further qualification and all other associated quality control sample results meet the acceptance criteria.

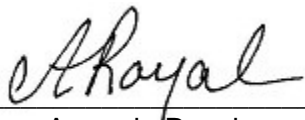
Batch B171659 (F4)

The second method blank (BLK2) recovers above the MRL and was reanalyzed as BLK5 for confirmation. Reanalysis as BLK5 confirms the high result and has been reported. All samples recover over ten times the hit in the highest blank, therefore no qualification is required. The duplicate (DUP1) performed on sample *SS-M-1500-051717* (1721026-03) does not meet RPD. A re-analysis of the source sample and corresponding QC set confirms the RPD and has been reported as DUP3. The source sample has been qualified **M**.

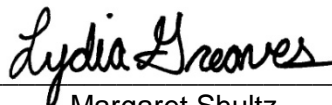
Batch B171660 (F5)

Method blanks BLK1 and BLK2 recover above the MRL and were reanalyzed for confirmation. The re-analyses of the BLKs (BLK5 and BLK6) confirmed high results. All samples recover over ten times the hit in the highest blank and therefore are not affected. The first duplicate (DUP1) recovered outside of RPD criteria and did not meet secondary criteria. The source sample *SS-M-1500-051717* (1721026-03) and its associated quality control were reanalyzed and confirmed the DUP RPD. The QC set DUP3 and PS3 have been reported and the source sample qualified **M**. The second post spike (PS2) was spiked below the concentration of the source sample. Therefore, post spike recoveries are not valid indicators of data quality. All data was reported without further qualification and all associated quality control sample results met the acceptance criteria.

We certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. BAL, an accredited laboratory, certifies that the reported results of all analyses for which BAL is NELAP accredited meet all NELAP requirements. For more details, please see the *Report Information* page in your report. Please feel free to contact us if you have any questions regarding this report.



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Report Information

Laboratory Accreditation

BAL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BAL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <http://www.brooksapplied.com/resources/certificates-permits/>. Results reported relate only to the samples listed in the report.

Field Quality Control Samples

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

Common Abbreviations

AR	as received	MS	matrix spike
BAL	Brooks Applied Labs	MSD	matrix spike duplicate
BLK	method blank	ND	non-detect
BS	blank spike	NR	non-reportable
CAL	calibration standard	N/C	not calculated
CCB	continuing calibration blank	PS	post preparation spike
CCV	continuing calibration verification	REC	percent recovery
COC	chain of custody record	RPD	relative percent difference
D	dissolved fraction	SCV	secondary calibration verification
DUP	duplicate	SOP	standard operating procedure
IBL	instrument blank	SRM	standard reference material
ICV	initial calibration verification	T	total fraction
MDL	method detection limit	TR	total recoverable fraction
MRL	method reporting limit		

Definition of Data Qualifiers

(Effective 9/23/09)

E	An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
H	Holding time and/or preservation requirements not met. Result is estimated.
J	Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
J-1	Estimated value. A full explanation is presented in the narrative.
J-M	Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
J-N	Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
M	Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
N	Spike recovery was not within acceptance criteria. Result is estimated.
R	Rejected, unusable value. A full explanation is presented in the narrative.
U	Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
X	Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Applied Labs, those found in the EPA SOW ILM03.0, Exhibit B, Section III, pg. B-18, and the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review; USEPA; January 2010. These supersede all previous qualifiers ever employed by BAL.



Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
SS-H-6000-051917	1721026-01	Soil	Sample	05/19/2017	05/25/2017
SS-H-96000-051917	1721026-02	Soil	Sample	05/19/2017	05/25/2017
SS-M-1500-051717	1721026-03	Soil	QC Sample	05/17/2017	05/25/2017
SS-H-CPx-10	1721026-04	Soil	Sample	06/09/2017	06/30/2017
SS-H-CPx-10-OPTI	1721026-05	Soil	Sample	06/09/2017	06/30/2017
SS-H-CPx-10-Solidified	1721026-06	Soil	QC Sample	06/09/2017	06/30/2017
SS-H-FB-15-OPTI	1721026-07	Soil	Sample	06/09/2017	06/30/2017
SS-H-FB-15-Solidified	1721026-08	Soil	Sample	06/09/2017	06/30/2017
SS-H-FB-CaO-10-Solidified	1721026-09	Soil	Sample	06/09/2017	06/30/2017
SS-H-S-25	1721026-10	Soil	Sample	06/09/2017	06/30/2017
SS-H-S-5	1721026-11	Soil	Sample	06/09/2017	06/30/2017
SS-M-CPx-5	1721026-12	Soil	Sample	06/09/2017	06/30/2017
SS-M-CPx-5-OPTI	1721026-13	Soil	Sample	06/09/2017	06/30/2017
SS-M-FB-5	1721026-14	Soil	Sample	06/09/2017	06/30/2017
SS-M-FB-CaO-5	1721026-15	Soil	Sample	06/09/2017	06/30/2017
SS-M-FB-CaO-5-OPTI	1721026-16	Soil	Sample	06/09/2017	06/30/2017



Batch Summary

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
%TS	Soil/Sediment	SM 2540G	07/10/2017	07/17/2017	B171661	N/A
Hg	Soil/Sediment	EPA 1631 Appendix	07/10/2017	07/14/2017	B171654	1700837
Hg(F0)	Soil/Sediment	SOP BAL-3900	07/11/2017	07/12/2017	B171685	1700825
Hg(F1)	Soil/Sediment	SOP BAL-3900	07/10/2017	07/14/2017	B171656	1700837
Hg(F1)	Soil/Sediment	SOP BAL-3900	07/10/2017	07/20/2017	B171656	1700862
Hg(F1)	Soil/Sediment	SOP BAL-3900	07/10/2017	07/24/2017	B171656	1700875
Hg(F1)	Soil/Sediment	SOP BAL-3900	07/10/2017	07/25/2017	B171656	1700882
Hg(F2)	Soil/Sediment	SOP BAL-3900	07/11/2017	07/14/2017	B171657	1700837
Hg(F2)	Soil/Sediment	SOP BAL-3900	07/11/2017	07/20/2017	B171657	1700862
Hg(F2)	Soil/Sediment	SOP BAL-3900	07/11/2017	07/22/2017	B171657	1700870
Hg(F3)	Soil/Sediment	SOP BAL-3900	07/12/2017	07/15/2017	B171658	1700837
Hg(F3)	Soil/Sediment	SOP BAL-3900	07/12/2017	07/20/2017	B171658	1700862
Hg(F4)	Soil/Sediment	SOP BAL-3900	07/13/2017	07/17/2017	B171659	1700846
Hg(F4)	Soil/Sediment	SOP BAL-3900	07/13/2017	07/20/2017	B171659	1700862
Hg(F5)	Soil/Sediment	SOP BAL-3900	07/14/2017	07/17/2017	B171660	1700846
Hg(F5)	Soil/Sediment	SOP BAL-3900	07/14/2017	07/20/2017	B171660	1700862



Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-H-6000-051917										
1721026-01	%TS	Soil	NA	92.01		0.01	0.03	%	B171661	N/A
1721026-01	Hg	Soil	dry	6990000		3060	10200	ng/g	B171654	1700837
1721026-01	Hg(F0)	Soil	dry	17300		49.6	49.6	ng/g	B171685	1700825
1721026-01	Hg(F1)	Soil	dry	23700		198	496	ng/g	B171656	1700837
1721026-01	Hg(F2)	Soil	dry	280000		24800	62000	ng/g	B171657	1700862
1721026-01	Hg(F3)	Soil	dry	405000		99200	248000	ng/g	B171658	1700862
1721026-01	Hg(F4)	Soil	dry	2940000		49600	124000	ng/g	B171659	1700846
1721026-01	Hg(F5)	Soil	dry	2610000		15900	39700	ng/g	B171660	1700846
SS-H-96000-051917										
1721026-02	%TS	Soil	NA	92.34		0.01	0.03	%	B171661	N/A
1721026-02	Hg	Soil	dry	6910000		2870	9560	ng/g	B171654	1700837
1721026-02	Hg(F0)	Soil	dry	21400		54.0	54.0	ng/g	B171685	1700825
1721026-02	Hg(F1)	Soil	dry	30900		216	540	ng/g	B171656	1700837
1721026-02	Hg(F2)	Soil	dry	335000		27000	67500	ng/g	B171657	1700862
1721026-02	Hg(F3)	Soil	dry	493000		108000	270000	ng/g	B171658	1700862
1721026-02	Hg(F4)	Soil	dry	3820000		54000	135000	ng/g	B171659	1700846
1721026-02	Hg(F5)	Soil	dry	3130000		17300	43200	ng/g	B171660	1700846
SS-H-CPx-10										
1721026-04	%TS	Soil	NA	87.94		0.01	0.03	%	B171661	N/A
1721026-04	Hg	Soil	dry	5990000		2860	9540	ng/g	B171654	1700837
1721026-04	Hg(F0)	Soil	dry	21700		55.7	55.7	ng/g	B171685	1700825
1721026-04	Hg(F1)	Soil	dry	44400		223	557	ng/g	B171656	1700862
1721026-04	Hg(F2)	Soil	dry	121	J	111	278	ng/g	B171657	1700862
1721026-04	Hg(F3)	Soil	dry	44800		891	2230	ng/g	B171658	1700837
1721026-04	Hg(F4)	Soil	dry	423000		55700	139000	ng/g	B171659	1700846
1721026-04	Hg(F5)	Soil	dry	5970000		178000	445000	ng/g	B171660	1700862



Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-H-CPx-10-OPTI										
1721026-05	%TS	Soil	NA	96.82		0.01	0.03	%	B171661	N/A
1721026-05	Hg	Soil	dry	7610000		2950	9840	ng/g	B171654	1700837
1721026-05	Hg(F0)	Soil	dry	262000	J-1	49.5	49.5	ng/g	B171685	1700825
1721026-05	Hg(F1)	Soil	dry	304000		19800	49500	ng/g	B171656	1700862
1721026-05	Hg(F2)	Soil	dry	368		98.9	247	ng/g	B171657	1700862
1721026-05	Hg(F3)	Soil	dry	32100		791	1980	ng/g	B171658	1700837
1721026-05	Hg(F4)	Soil	dry	3040000		49500	124000	ng/g	B171659	1700846
1721026-05	Hg(F5)	Soil	dry	2890000		15800	39600	ng/g	B171660	1700862
SS-H-CPx-10-Solidified										
1721026-06	%TS	Soil	NA	92.61		0.01	0.03	%	B171661	N/A
1721026-06	Hg	Soil	dry	5340000		3220	10700	ng/g	B171654	1700837
1721026-06	Hg(F0)	Soil	dry	170		46.3	46.3	ng/g	B171685	1700825
1721026-06	Hg(F1)	Soil	dry	32.1	N	0.19	0.46	ng/g	B171656	1700875
1721026-06	Hg(F2)	Soil	dry	23200	M	185	463	ng/g	B171657	1700862
1721026-06	Hg(F3)	Soil	dry	9610	M	926	2320	ng/g	B171658	1700862
1721026-06	Hg(F4)	Soil	dry	2820000		46300	116000	ng/g	B171659	1700846
1721026-06	Hg(F5)	Soil	dry	2390000		14800	37100	ng/g	B171660	1700846
SS-H-FB-15-OPTI										
1721026-07	%TS	Soil	NA	95.50		0.01	0.03	%	B171661	N/A
1721026-07	Hg	Soil	dry	4840000		2630	8780	ng/g	B171654	1700837
1721026-07	Hg(F0)	Soil	dry	609		46.3	46.3	ng/g	B171685	1700825
1721026-07	Hg(F1)	Soil	dry	1110		23.2	57.9	ng/g	B171656	1700882
1721026-07	Hg(F2)	Soil	dry	871		92.7	232	ng/g	B171657	1700862
1721026-07	Hg(F3)	Soil	dry	21800		741	1850	ng/g	B171658	1700837
1721026-07	Hg(F4)	Soil	dry	2770000		46300	116000	ng/g	B171659	1700846
1721026-07	Hg(F5)	Soil	dry	1140000		14800	37100	ng/g	B171660	1700846



Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-H-FB-15-Solidified										
1721026-08	%TS	Soil	NA	85.41		0.01	0.03	%	B171661	N/A
1721026-08	Hg	Soil	dry	8830000		3040	10100	ng/g	B171654	1700837
1721026-08	Hg(F0)	Soil	dry	301000	J-1	51.4	51.4	ng/g	B171685	1700825
1721026-08	Hg(F1)	Soil	dry	420000		25700	64200	ng/g	B171656	1700862
1721026-08	Hg(F2)	Soil	dry	461		103	257	ng/g	B171657	1700862
1721026-08	Hg(F3)	Soil	dry	181000		822	2050	ng/g	B171658	1700837
1721026-08	Hg(F4)	Soil	dry	2770000		51400	128000	ng/g	B171659	1700846
1721026-08	Hg(F5)	Soil	dry	2670000		16400	41100	ng/g	B171660	1700846
SS-H-FB-CaO-10-Solidified										
1721026-09	%TS	Soil	NA	85.24		0.01	0.03	%	B171661	N/A
1721026-09	Hg	Soil	dry	4120000		3430	11400	ng/g	B171654	1700837
1721026-09	Hg(F0)	Soil	dry	336000	J-1	55.2	55.2	ng/g	B171685	1700825
1721026-09	Hg(F1)	Soil	dry	354000		27600	69000	ng/g	B171656	1700862
1721026-09	Hg(F2)	Soil	dry	39200		221	552	ng/g	B171657	1700837
1721026-09	Hg(F3)	Soil	dry	6480		883	2210	ng/g	B171658	1700837
1721026-09	Hg(F4)	Soil	dry	949000		55200	138000	ng/g	B171659	1700846
1721026-09	Hg(F5)	Soil	dry	1870000		17700	44200	ng/g	B171660	1700846
SS-H-S-25										
1721026-10	%TS	Soil	NA	93.02		0.01	0.03	%	B171661	N/A
1721026-10	Hg	Soil	dry	7830000		3230	10800	ng/g	B171654	1700837
1721026-10	Hg(F0)	Soil	dry	7530		47.7	47.7	ng/g	B171685	1700825
1721026-10	Hg(F1)	Soil	dry	8870		191	477	ng/g	B171656	1700862
1721026-10	Hg(F2)	Soil	dry	49400		191	477	ng/g	B171657	1700837
1721026-10	Hg(F3)	Soil	dry	86500		763	1910	ng/g	B171658	1700837
1721026-10	Hg(F4)	Soil	dry	3230000		47700	119000	ng/g	B171659	1700846
1721026-10	Hg(F5)	Soil	dry	1970000		15300	38100	ng/g	B171660	1700846



Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-H-S-5										
1721026-11	%TS	Soil	NA	91.83		0.01	0.03	%	B171661	N/A
1721026-11	Hg	Soil	dry	6280000		2970	9900	ng/g	B171654	1700837
1721026-11	Hg(F0)	Soil	dry	15400		54.0	54.0	ng/g	B171685	1700825
1721026-11	Hg(F1)	Soil	dry	11400		216	540	ng/g	B171656	1700837
1721026-11	Hg(F2)	Soil	dry	79100		216	540	ng/g	B171657	1700837
1721026-11	Hg(F3)	Soil	dry	9500		864	2160	ng/g	B171658	1700837
1721026-11	Hg(F4)	Soil	dry	3920000		54000	135000	ng/g	B171659	1700846
1721026-11	Hg(F5)	Soil	dry	2320000		17300	43200	ng/g	B171660	1700846
SS-M-1500-051717										
1721026-03	%TS	Soil	NA	93.01		0.01	0.03	%	B171661	N/A
1721026-03	Hg	Soil	dry	2300000		2880	9610	ng/g	B171654	1700837
1721026-03	Hg(F0)	Soil	dry	7580	M	50.3	50.3	ng/g	B171685	1700825
1721026-03	Hg(F1)	Soil	dry	12900	M	201	503	ng/g	B171656	1700862
1721026-03	Hg(F2)	Soil	dry	5660	M	201	503	ng/g	B171657	1700870
1721026-03	Hg(F3)	Soil	dry	20300	M	804	2010	ng/g	B171658	1700862
1721026-03	Hg(F4)	Soil	dry	1080000	M	50300	126000	ng/g	B171659	1700862
1721026-03	Hg(F5)	Soil	dry	2940000	M	16100	40200	ng/g	B171660	1700862
SS-M-CPx-5										
1721026-12	%TS	Soil	NA	90.59		0.01	0.03	%	B171661	N/A
1721026-12	Hg	Soil	dry	1840000		2850	9490	ng/g	B171654	1700837
1721026-12	Hg(F0)	Soil	dry	75200	J-1	46.3	46.3	ng/g	B171685	1700825
1721026-12	Hg(F1)	Soil	dry	10400		185	463	ng/g	B171656	1700837
1721026-12	Hg(F2)	Soil	dry	176		23.1	57.8	ng/g	B171657	1700862
1721026-12	Hg(F3)	Soil	dry	27000		740	1850	ng/g	B171658	1700837
1721026-12	Hg(F4)	Soil	dry	363000		46300	116000	ng/g	B171659	1700846
1721026-12	Hg(F5)	Soil	dry	1630000		14800	37000	ng/g	B171660	1700846



Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-M-CPx-5-OPTI										
1721026-13	%TS	Soil	NA	98.42		0.01	0.03	%	B171661	N/A
1721026-13	Hg	Soil	dry	2830000		2880	9610	ng/g	B171654	1700837
1721026-13	Hg(F0)	Soil	dry	150		48.8	48.8	ng/g	B171685	1700825
1721026-13	Hg(F1)	Soil	dry	742		97.6	244	ng/g	B171656	1700862
1721026-13	Hg(F2)	Soil	dry	255		24.4	61.0	ng/g	B171657	1700862
1721026-13	Hg(F3)	Soil	dry	12300		781	1950	ng/g	B171658	1700837
1721026-13	Hg(F4)	Soil	dry	599000		48800	122000	ng/g	B171659	1700846
1721026-13	Hg(F5)	Soil	dry	842000		15600	39100	ng/g	B171660	1700846
SS-M-FB-5										
1721026-14	%TS	Soil	NA	92.22		0.01	0.03	%	B171661	N/A
1721026-14	Hg	Soil	dry	2740000		2940	9810	ng/g	B171654	1700837
1721026-14	Hg(F0)	Soil	dry	233		49.6	49.6	ng/g	B171685	1700825
1721026-14	Hg(F1)	Soil	dry	4260		198	496	ng/g	B171656	1700837
1721026-14	Hg(F2)	Soil	dry	259		24.8	62.0	ng/g	B171657	1700862
1721026-14	Hg(F3)	Soil	dry	6380		794	1980	ng/g	B171658	1700837
1721026-14	Hg(F4)	Soil	dry	528000		49600	124000	ng/g	B171659	1700846
1721026-14	Hg(F5)	Soil	dry	1200000		15900	39700	ng/g	B171660	1700846
SS-M-FB-CaO-5										
1721026-15	%TS	Soil	NA	93.57		0.01	0.03	%	B171661	N/A
1721026-15	Hg	Soil	dry	1790000		3080	10300	ng/g	B171654	1700837
1721026-15	Hg(F0)	Soil	dry	2890		48.4	48.4	ng/g	B171685	1700825
1721026-15	Hg(F1)	Soil	dry	4550		194	484	ng/g	B171656	1700837
1721026-15	Hg(F2)	Soil	dry	310		24.2	60.5	ng/g	B171657	1700862
1721026-15	Hg(F3)	Soil	dry	5290		775	1940	ng/g	B171658	1700837
1721026-15	Hg(F4)	Soil	dry	209000		48400	121000	ng/g	B171659	1700846
1721026-15	Hg(F5)	Soil	dry	1520000		15500	38700	ng/g	B171660	1700846

Project ID: KAT-SC1701
PM: Amanda Royal -



BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Sample Results

Sample	Analyte	Report Matrix	Basis	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
SS-M-FB-CaO-5-OPTI										
1721026-16	%TS	Soil	NA	99.11		0.01	0.03	%	B171661	N/A
1721026-16	Hg	Soil	dry	4070000		2640	8800	ng/g	B171654	1700837
1721026-16	Hg(F0)	Soil	dry	68.8		46.0	46.0	ng/g	B171685	1700825
1721026-16	Hg(F1)	Soil	dry	557		91.9	230	ng/g	B171656	1700862
1721026-16	Hg(F2)	Soil	dry	120		23.0	57.4	ng/g	B171657	1700862
1721026-16	Hg(F3)	Soil	dry	5580		735	1840	ng/g	B171658	1700837
1721026-16	Hg(F4)	Soil	dry	519000		46000	115000	ng/g	B171659	1700846
1721026-16	Hg(F5)	Soil	dry	899000		14700	36800	ng/g	B171660	1700846



Accuracy & Precision Summary

Batch: B171654
Lab Matrix: Soil/Sediment
Method: EPA 1631 Appendix

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171654-SRM1	Standard Reference Material (1529016, MESS-4)						
	Hg		80.00	93.23	ng/g	117% 75-125	
B171654-SRM2	Standard Reference Material (1529016, MESS-4)						
	Hg		80.00	74.54	ng/g	93% 75-125	
B171654-DUP2	Duplicate (1721026-03)						
	Hg	2299000		2098000	ng/g		9% 30
B171654-MS2	Matrix Spike (1721026-03)						
	Hg	2299000	19660	2808000	ng/g	NR 70-130	
B171654-MSD2	Matrix Spike Duplicate (1721026-03)						
	Hg	2299000	18720	2159000	ng/g	NR 70-130	N/C 30
B171654-DUP3	Duplicate (1721026-06)						
	Hg	5339000		6142000	ng/g		14% 30
B171654-MS3	Matrix Spike (1721026-06)						
	Hg	5339000	21570	6184000	ng/g	NR 70-130	
B171654-MSD3	Matrix Spike Duplicate (1721026-06)						
	Hg	5339000	21210	6493000	ng/g	NR 70-130	N/C 30



Accuracy & Precision Summary

Batch: B171656
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171656-SRM1	Standard Reference Material (0721002, NIST 2710) Hg(F1)		203.0	190.2	ng/g	94% 75-125	
B171656-SRM2	Standard Reference Material (1349004, Red HgS Spiked Kaolin) Hg(F1)		30.00	21.33	ng/g	71% 75-125	
B171656-SRM3	Standard Reference Material (1349005, Hg0 Spiked Kaolin) Hg(F1)		77900	765000	ng/g	982% 75-125	
B171656-SRM4	Standard Reference Material (1349006, HgCl2 Spiked Kaolin) Hg(F1)		1434000	764800	ng/g	53% 75-125	
B171656-DUP3	Duplicate, (1721026-03) Hg(F1)	12910		5341	ng/g		83% 35
B171656-PS3	Post Spike, (1721026-03) Hg(F1)	12910	25130	38030	ng/g	100% 77-123	
B171656-DUP6	Duplicate, (1721026-06) Hg(F1)	32.11		33.18	ng/g		3% 35
B171656-PS6	Post Spike, (1721026-06) Hg(F1)	32.11	23.16	49.64	ng/g	76% 77-123	



Accuracy & Precision Summary

Batch: B171657
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171657-SRM1	Standard Reference Material (0721002, NIST 2710) Hg(F2)		28.10	88.25	ng/g	314% 75-125	
B171657-SRM3	Standard Reference Material (1349005, Hg0 Spiked Kaolin) Hg(F2)		76600	100800	ng/g	132% 75-125	
B171657-SRM5	Standard Reference Material (1349004, Red HgS Spiked Kaolin) Hg(F2)		18.00	27.94	ng/g	155% 75-125	
B171657-SRM6	Standard Reference Material (1349006, HgCl2 Spiked Kaolin) Hg(F2)		216000	192000	ng/g	89% 75-125	
B171657-DUP5	Duplicate, (1721026-03) Hg(F2)	5657		288.7	ng/g		181% 35
B171657-PS5	Post Spike, (1721026-03) Hg(F2)	5657	25130	30260	ng/g	98% 77-123	
B171657-DUP4	Duplicate, (1721026-06) Hg(F2)	23180		808.8	ng/g		187% 35
B171657-PS4	Post Spike, (1721026-06) Hg(F2)	23180	23160	47340	ng/g	104% 77-123	



Accuracy & Precision Summary

Batch: B171658
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171658-SRM1	Standard Reference Material (0721002, NIST 2710) Hg(F3)		704.0	733.1	ng/g	104% 75-125	
B171658-SRM3	Standard Reference Material (1349005, Hg0 Spiked Kaolin) Hg(F3)		393000	578000	ng/g	147% 75-125	
B171658-SRM5	Standard Reference Material (1349004, Red HgS Spiked Kaolin) Hg(F3)		8.000	84840	ng/g	60000% 75-125	
B171658-SRM6	Standard Reference Material (1349006, HgCl2 Spiked Kaolin) Hg(F3)		79500	280300	ng/g	353% 75-125	
B171658-DUP3	Duplicate, (1721026-03) Hg(F3)	20310		5724	ng/g		112% 35
B171658-PS3	Post Spike, (1721026-03) Hg(F3)	20310	2513	52160	ng/g	1270% 77-123	
B171658-DUP4	Duplicate, (1721026-06) Hg(F3)	9611		23250	ng/g		83% 35
B171658-PS4	Post Spike, (1721026-06) Hg(F3)	9611	2316	37600	ng/g	1210% 77-123	



Accuracy & Precision Summary

Batch: B171659
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171659-SRM1	Standard Reference Material (0721002, NIST 2710) Hg(F4)		12930	23010	ng/g	178% 75-125	
B171659-SRM2	Standard Reference Material (1349004, Red HgS Spiked Kaolin) Hg(F4)		8300	36440	ng/g	439% 75-125	
B171659-SRM3	Standard Reference Material (1349005, Hg0 Spiked Kaolin) Hg(F4)		5238000	4187000	ng/g	80% 75-125	
B171659-SRM4	Standard Reference Material (1349006, HgCl2 Spiked Kaolin) Hg(F4)		120000	187300	ng/g	156% 75-125	
B171659-DUP3	Duplicate, (1721026-03) Hg(F4)	1084000		506800	ng/g		73% 35
B171659-PS3	Post Spike, (1721026-03) Hg(F4)	1084000	3141000	4121000	ng/g	97% 77-123	
B171659-DUP2	Duplicate, (1721026-06) Hg(F4)	2820000		2993000	ng/g		6% 35
B171659-PS2	Post Spike, (1721026-06) Hg(F4)	2820000	2895000	5870000	ng/g	105% 77-123	



Accuracy & Precision Summary

Batch: B171660
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171660-SRM1	Standard Reference Material (0721002, NIST 2710) Hg(F5)		18150	12520	ng/g	69% 75-125	
B171660-SRM2	Standard Reference Material (1349004, Red HgS Spiked Kaolin) Hg(F5)		2013000	1623000	ng/g	81% 75-125	
B171660-SRM3	Standard Reference Material (1349005, Hg0 Spiked Kaolin) Hg(F5)		16600	31110	ng/g	187% 75-125	
B171660-SRM4	Standard Reference Material (1349006, HgCl2 Spiked Kaolin) Hg(F5)		20600	30350	ng/g	147% 75-125	
B171660-DUP3	Duplicate, (1721026-03) Hg(F5)	2939000		1279000	ng/g		79% 35
B171660-PS3	Post Spike, (1721026-03) Hg(F5)	2939000	804000	3769000	ng/g	103% 77-123	
B171660-DUP2	Duplicate, (1721026-06) Hg(F5)	2395000		2985000	ng/g		22% 35
B171660-PS2	Post Spike, (1721026-06) Hg(F5)	2395000	741100	3353000	ng/g	129% 77-123	

Project ID: KAT-SC1701
PM: Amanda Royal -



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Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Accuracy & Precision Summary

Batch: B171661
Lab Matrix: Soil/Sediment
Method: SM 2540G

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171661-DUP1	Duplicate, (1721026-03) %TS	93.01		93.63	%		0.7% 15
B171661-DUP2	Duplicate, (1721026-06) %TS	92.61		92.40	%		0.2% 15

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PM: Amanda Royal -



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Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Accuracy & Precision Summary

Batch: B171685
Lab Matrix: Soil/Sediment
Method: SOP BAL-3900

Sample	Analyte	Native	Spike	Result	Units	REC & Limits	RPD & Limits
B171685-DUP1	Duplicate, (1721026-03) Hg(F0)	7583		863.1	ng/g		159% 35
B171685-DUP2	Duplicate, (1721026-06) Hg(F0)	169.9		145.0	ng/g		16% 35

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PM: Amanda Royal -



BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171654
Matrix: Soil/Sediment
Method: EPA 1631 Appendix
Analyte: Hg

Sample	Result	Units		
B171654-BLK1	0.047	ng/g		
B171654-BLK2	0.035	ng/g		
B171654-BLK3	0.035	ng/g		
B171654-BLK4	0.028	ng/g		
Average: 0.036		Standard Deviation: 0.008	MDL: 0.150	
Limit: 0.300		Limit: 0.100	MRL: 0.500	

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Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171656
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F1)

Sample	Result	Units		
B171656-BLK2	0.30	ng/g		
B171656-BLK3	0.18	ng/g		
B171656-BLK4	0.14	ng/g		
Average: 0.21		Standard Deviation: 0.08		MDL: 0.20
Limit: 0.40		Limit: 0.13		MRL: 0.50

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PM: Amanda Royal -



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Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171657
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F2)

Sample	Result	Units		
B171657-BLK2	0.36	ng/g		
B171657-BLK3	0.12	ng/g		
B171657-BLK4	0.19	ng/g		
Average: 0.22			Standard Deviation: 0.12	MDL: 0.20
Limit: 0.40			Limit: 0.13	MRL: 0.50

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BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171658
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F3)

Sample	Result	Units		
B171658-BLK1	2.74	ng/g		
B171658-BLK2	1.99	ng/g		
B171658-BLK3	0.28	ng/g		
Average: 1.67			Standard Deviation: 1.26	MDL: 2.00
Limit: 4.00			Limit: 1.33	MRL: 5.00

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PM: Amanda Royal -



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Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171659
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F4)

Sample	Result	Units		
B171659-BLK1	3.25	ng/g		
B171659-BLK3	0.52	ng/g		
B171659-BLK4	3.31	ng/g		
B171659-BLK5	6.08	ng/g		
Average: 3.29		Standard Deviation: 2.27		MDL: 2.00
Limit: 4.00		Limit: 1.33		MRL: 5.00

Project ID: KAT-SC1701
PM: Amanda Royal -



BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171660
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F5)

Sample	Result	Units
B171660-BLK3	2.06	ng/g
B171660-BLK4	1.13	ng/g
B171660-BLK5	5.96	ng/g
B171660-BLK6	11.9	ng/g

Average: 5.27
Limit: 4.00

Standard Deviation: 4.90
Limit: 1.33

MDL: 2.00
MRL: 5.00

Project ID: KAT-SC1701
PM: Amanda Royal -



BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171661
Matrix: Soil/Sediment
Method: SM 2540G
Analyte: %TS

Sample	Result	Units
B171661-BLK1	0.01	%
B171661-BLK2	-0.009	%

Average: 0.00
Limit: 0.03

MDL: 0.01
MRL: 0.03

Project ID: KAT-SC1701
PM: Amanda Royal -



BAL Report 1721026, Rev. 1
Client PM: Allison Harbottle
Client Project: LCP Chemical Superfund Site

Method Blanks & Reporting Limits

Batch: B171685
Matrix: Soil/Sediment
Method: SOP BAL-3900
Analyte: Hg(F0)

Sample	Result	Units		
B171685-BLK1	3.58	ng/g		
B171685-BLK2	22.0	ng/g		
B171685-BLK3	4.59	ng/g		
B171685-BLK4	4.09	ng/g		
Average: 8.57		Standard Deviation: 8.98	MDL: 50.0	
Limit: 100.00		Limit: 33.33	MRL: 50.0	

Appendix D

CDM Smith Geotechnical Laboratory Report

Unconfined Compressive Strength (ASTM D2166)

Test Performed by : AS
Test Date : 8/10/17

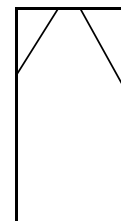
Soil Type : Soil CementPreparation Method: Smoothed ends

Loading Rate (in/min) :	0.05
Dial Rate :	5.8
Strain Rate (%/min)	1.25
Strain at Failure (%) :	2.44
U. C. Strength (psi) :	610.1
Shear Strength (psi):	305.0

The graph plots Compressive Stress (psi) on the y-axis against Axial Strain (%) on the x-axis. The y-axis ranges from 0.0 to 350.0 in increments of 50.0. The x-axis ranges from 0.0 to 4.0 in increments of 1.0. The data points are connected by a solid blue line, showing a non-linear relationship that peaks and then slightly declines.

Axial Strain (%)	Compressive Stress (psi)
0.0	0.0
0.2	15.0
0.4	45.0
0.6	105.0
0.8	195.0
1.0	255.0
1.2	285.0
1.4	300.0
1.6	295.0
1.8	300.0
2.0	290.0
2.2	285.0

Failure Sketch



Remarks: None.

CDM Smith

Geotechnical Engineering Laboratory

Hydraulic Conductivity Using Flexible Wall Permeameter (ASTM D5084)

Client: USEPA/USACE
Project Name: LCP Chemical
Project Location: Linden, NJ
Project Number: 51147-113481
Sample Number: SS-H-CPx-10
Sample Date: 6/9/2017
Depth (ft):
Sample Description: Solidified
Test Type: ASTM D5084

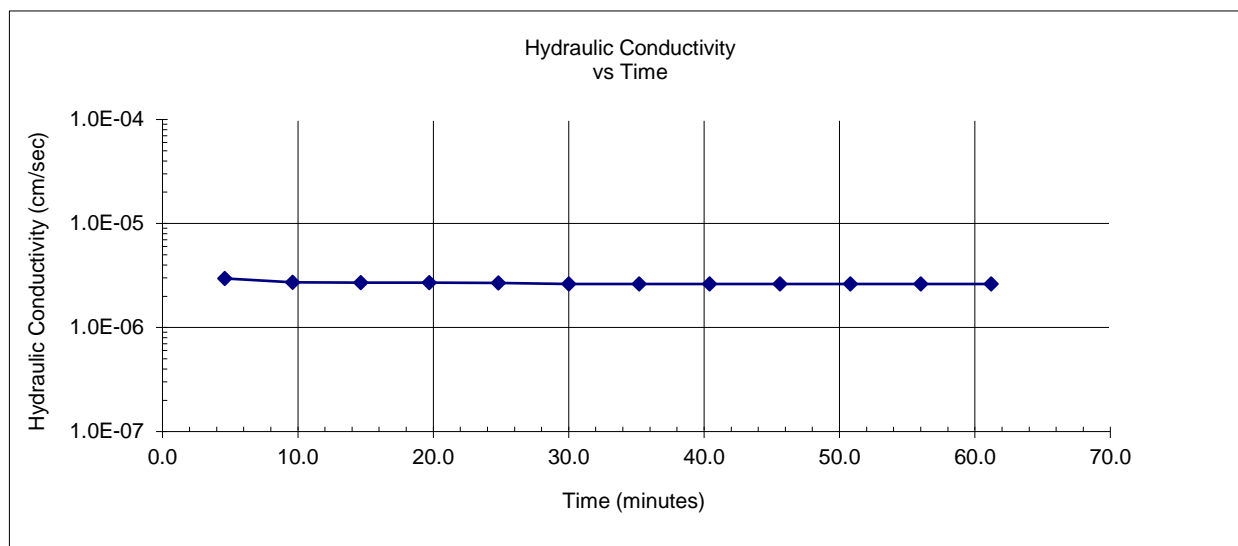
Tested by: ACS
Checked by: MBP
Start Test Date: 8/7/2017
Permeant Fluid: De-aired water
Sample Preparation
Procedures: Ends smoothed for testing

Sample Characteristics	Initial	Final
Avg. length of specimen (in)	1.85	1.85
Avg. dia. of specimen (in)	1.82	1.82
Area (sq in)	2.60	2.60
Volume (cubic in)	4.80	4.80
Moist mass (g)	127.1	148.1
Moist density (pcf)	101.0	117.6
Moisture content (%)	12.3	30.8
Dry density (pcf)	89.9	89.9
Specific gravity (assumed)	2.65	2.65
Void ratio	0.84	0.84

Test Specifications	
B-Value (%):	100.0
Consolidation stress (psi):	5.0
Gradient (in/in):	34.3
Cell pressure (psi):	85.0
Head pressure (psi):	82.0
Tail pressure (psi):	80.0
Max effective stress (psi):	5.0
Min effective stress (psi):	3.0

Comments: Sample was divided vertically into quarters.
No observed anomalies (ie rocks, voids, etc.).

Hydraulic Conductivity at 20 °C = **2.62E-06** cm/sec
Average of last 6 data points



Unconfined Compressive Strength (ASTM D2166)

Test Performed by : AS
Test Date : 8/10/17

Soil Type : Soil Cement

Preparation Method: Smoothed ends

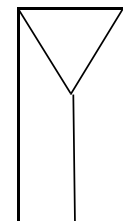
Notes: 1" down from top, 0.4"
dia. hole, 0.8" deep

Loading Rate (in/min) :	0.05
Dial Rate :	5.8
Strain Rate (%/min)	1.24
Strain at Failure (%) :	0.96
U. C. Strength (psi) :	113.5
Shear Strength (psi):	56.7

The graph plots Compressive Stress (psi) on the y-axis (0.0 to 120.0) against Axial Strain (%) on the x-axis (0.0 to 4.0). The data points are connected by a solid blue line. The curve shows a sharp initial rise to a peak stress of approximately 115 psi at 0.8% strain, followed by a significant drop to about 85 psi at 1.2% strain. After this, the stress levels off slightly before gradually decreasing to approximately 45 psi at 3.8% strain.

Axial Strain (%)	Compressive Stress (psi)
0.0	0.0
0.2	7.0
0.4	27.0
0.6	73.0
0.8	112.0
0.9	114.0
1.2	85.0
1.4	75.0
1.6	72.0
1.8	72.0
2.0	73.0
2.2	70.0
2.4	67.0
2.6	66.0
2.8	68.0
3.0	56.0
3.2	54.0
3.8	45.0

Failure Sketch



Remarks: None.

CDM Smith

Geotechnical Engineering Laboratory

Hydraulic Conductivity Using Flexible Wall Permeameter (ASTM D5084)

Client: USEPA/USACE
Project Name: LCP Chemical
Project Location: Linden, NJ
Project Number: 51147-113481
Sample Number: SS-H-FB-15
Sample Date: 6/9/2017
Depth (ft):
Sample Description: Solidified
Test Type: ASTM D5084

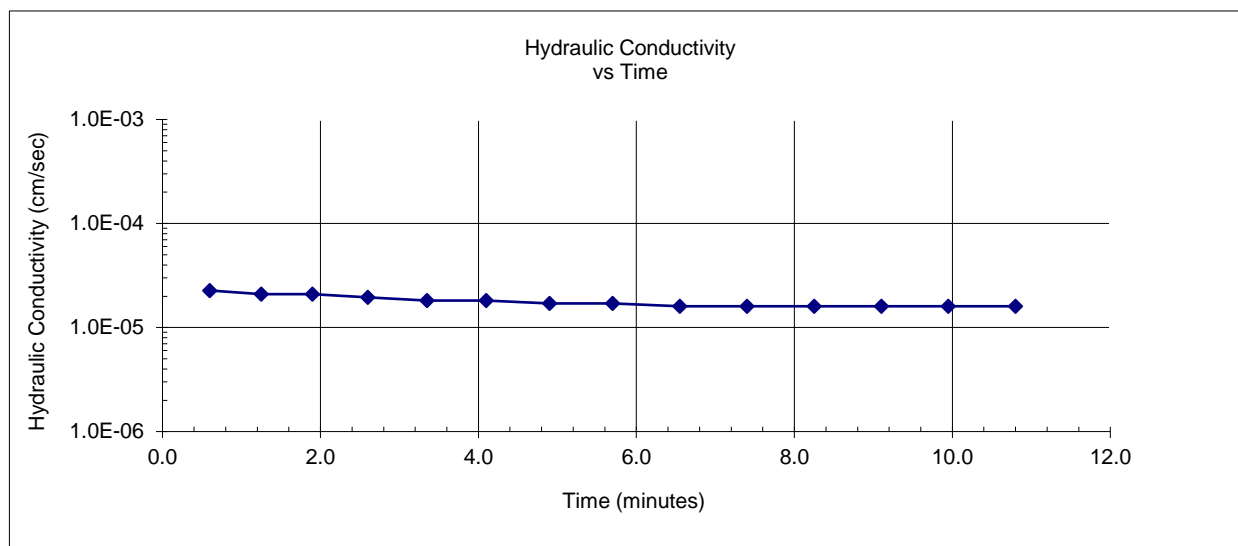
Tested by: ACS
Checked by: MBP
Start Test Date: 8/8/2017
Permeant Fluid: De-aired water
Sample Preparation
Procedures: Ends smoothed for testing

Sample Characteristics	Initial	Final
Avg. length of specimen (in)	1.80	1.80
Avg. dia. of specimen (in)	1.84	1.84
Area (sq in)	2.66	2.66
Volume (cubic in)	4.79	4.79
Moist mass (g)	126.3	149.8
Moist density (pcf)	100.4	119.2
Moisture content (%)	6.7	26.6
Dry density (pcf)	94.1	94.1
Specific gravity (assumed)	2.65	2.65
Void ratio	0.76	0.76

Test Specifications	
B-Value (%):	100.0
Consolidation stress (psi):	5.0
Gradient (in/in):	33.6
Cell pressure (psi):	85.0
Head pressure (psi):	82.0
Tail pressure (psi):	80.0
Max effective stress (psi):	5.0
Min effective stress (psi):	3.0

Comments: Sample was divided vertically into quarters.
No observed anomalies (ie rocks, voids, etc.).

Hydraulic Conductivity at 20 °C = **1.60E-05** cm/sec
Average of last 6 data points



Unconfined Compressive Strength (ASTM D2166)

Test Performed by : AS
Test Date : 8/10/17

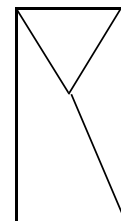
Soil Type : Soil CementPreparation Method: Smoothed ends

Loading Rate (in/min) :	0.05
Dial Rate :	5.8
Strain Rate (%/min)	1.24
Strain at Failure (%) :	1.47
U. C. Strength (psi) :	583.8
Shear Strength (psi):	291.9

The graph illustrates the relationship between Compressive Stress (psi) and Axial Strain (%) for a material. The stress starts at 0 psi at 0% strain, rises to a peak of approximately 580 psi at 1.5% strain, and then decreases to about 240 psi at 2.0% strain. For strains greater than 2.5%, the stress levels off around 80-90 psi.

Axial Strain (%)	Compressive Stress (psi)
0.0	0.0
0.2	10.0
0.4	20.0
0.6	60.0
0.8	190.0
1.0	340.0
1.2	470.0
1.4	550.0
1.5	580.0
1.7	520.0
2.0	240.0
2.2	200.0
2.4	120.0
2.6	90.0
2.8	85.0
3.0	85.0
3.2	80.0
3.6	85.0

Failure Sketch



Remarks: None.

CDM Smith

Geotechnical Engineering Laboratory

Hydraulic Conductivity Using Flexible Wall Permeameter (ASTM D5084)

Client: USEPA/USACE
Project Name: LCP Chemical
Project Location: Linden, NJ
Project Number: 51147-113481
Sample Number: SS-H-CaO-FB-10
Sample Date: 6/9/2017
Depth (ft):
Sample Description: Solidified
Test Type: ASTM D5084

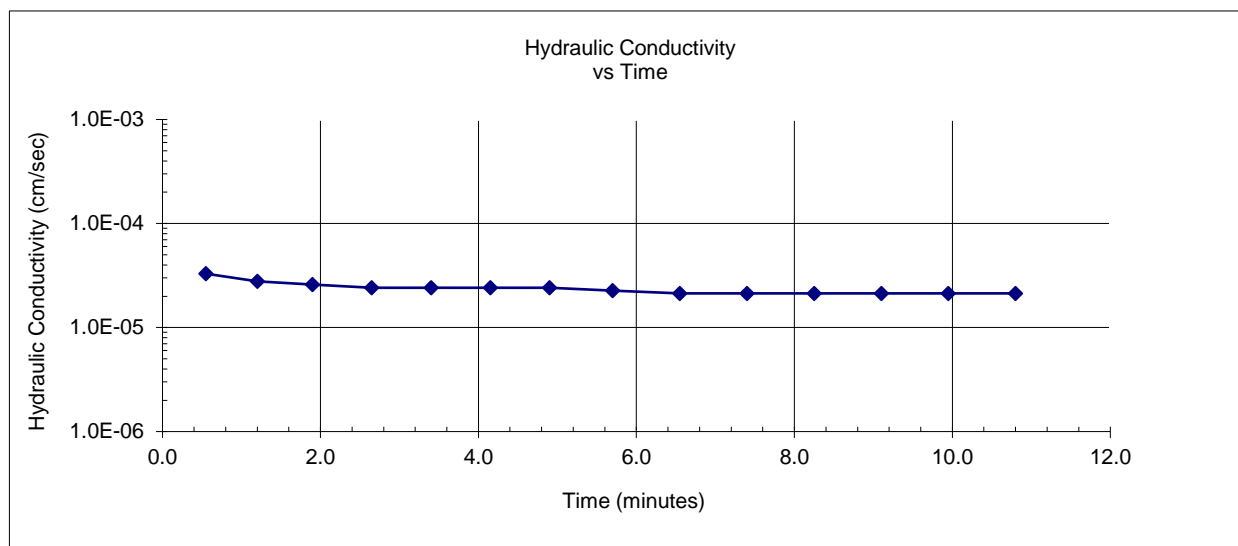
Tested by: ACS
Checked by: MBP
Start Test Date: 8/8/2017
Permeant Fluid: De-aired water
Sample Preparation
Procedures: Ends smoothed for testing

Sample Characteristics	Initial	Final
Avg. length of specimen (in)	2.35	2.35
Avg. dia. of specimen (in)	1.81	1.81
Area (sq in)	2.57	2.57
Volume (cubic in)	6.03	6.03
Moist mass (g)	140.5	182.2
Moist density (pcf)	88.8	115.2
Moisture content (%)	5.0	36.2
Dry density (pcf)	84.6	84.6
Specific gravity (assumed)	2.65	2.65
Void ratio	0.96	0.96

Test Specifications	
B-Value (%):	100.0
Consolidation stress (psi):	5.0
Gradient (in/in):	26.2
Cell pressure (psi):	85.0
Head pressure (psi):	82.0
Tail pressure (psi):	80.0
Max effective stress (psi):	5.0
Min effective stress (psi):	3.0

Comments: Sample was divided vertically into quarters.
No observed anomalies (ie rocks, voids, etc.).

Hydraulic Conductivity at 20 °C = **2.13E-05** cm/sec
Average of last 6 data points



Appendix E

Bench Scale Study Data Usability Assessment Report

REPORT

LCP Chemicals, Inc. Superfund Site Linden, New Jersey

Data Usability Report No. 2
(Bench Scale Study)

December 2017



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Appendix

Appendix A Data Validation Reports

Acronyms

%	percent
%D	Percent Difference
%R	Percent Recovery
AES	atomic emission spectrometer
CLP	Contract Laboratory Program
DQI	data quality indicator
DQO	data quality objective
DTL	CDM Smith Denver, Colorado Treatability Laboratory
DUAR	data usability assessment review
EPA	Environmental Protection Agency
Hg ⁰	elemental mercury
HgCl ₂	mercuric chloride
HgS	mercuric sulfide
ICP	inductively coupled plasma
LCS/LCSD	laboratory control sample/laboratory control sample duplicate
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PDI	Pre-Design Investigation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
R	recovery
RCRA	Resource Conservation and Recovery Act
RL	reporting limit
RPD	relative percent difference
RSD	relative standard deviation
SDG	sample delivery group
SDL	semi-dynamic leaching
SGC	Studio Geochemica
SM	standard method
SOP	standard operating procedure
SPLP	synthetic precipitation leaching procedure
SRM	standard reference material
SSE	selective sequential extraction

Section 1

Data Usability Assessment Review

The purpose of this data usability assessment review (DUAR) is to determine the usability of the data collected at the LCP Chemicals Inc. Superfund Site (the Site) located in Linden, Union County, New Jersey and to determine whether the sample results meet the data quality objectives (DQOs) outlined in the project Final Quality Assurance Project Plan (QAPP) (CDM Smith 2017).

The samples addressed in this DUAR were collected for the bench scale treatability study performed at the CDM Smith Denver, Colorado Treatability Laboratory (DTL). Samples were collected to determine the specific additives and application methods for in situ stabilization/solidification of the elemental mercury (Hg^0) at the Site. Samples were collected April 11, 2017 through April 14, 2017. These samples were prepared (dried, composited and subsampled) at the CDM Smith DTL and then submitted to two United States Environmental Protection Agency (EPA) National Contract Laboratory Program (CLP) laboratories for analyses of mercury and metal analytes. Leachates (synthetic precipitation leaching procedure and semi-dynamic leaching [SPLP and SDL] methods) were also generated at the CDM Smith DTL and submitted to the EPA CLP laboratories for analyses of mercury and Resource Conservation and Recovery Act (RCRA) metals.

A subset of the prepared samples from the CDM Smith DTL were also sent to a subcontract laboratory, Brooks Applied Laboratory, for confirmation analyses of the treatability study mercury and mercury sulfide analyses performed by the CDM Smith DTL.

Laboratories providing analytical services included EPA CLP Laboratories, Bonner Analytical Testing Company and Chemtech Consulting Group, and CDM Smith subcontractors, Brooks Applied Laboratory lower tiered by Katahdin Analytical Services. Analyses are identified in Table 1-1.

This report includes a summary of the validation performed on the samples and an overall assessment of the data quality and usability. EPA and CDM Smith performed the data validation in accordance with the QAPP requirements.

CDM Smith conducted data validation on the mercury and mercury speciation data analyzed by Brooks Applied Laboratory. The EPA Region 2 data validators and their contractors validated the samples analyzed by the EPA CLP laboratories. CDM Smith data validation specialists also reviewed the quality control (QC) parameters such as calibration standards, sand blanks and Hg^0 versus black and red cinnabar reagent grade mercuric sulfide (HgS) associated with the CDM Smith DTL bench scale treatability sample preparation and analysis procedures and results.

1.1 Usability Summary

Over 90 percent (%) of the soil and leachate data samples validated and reported herein are suitable for the intended use as stated in the QAPP and can be used for the Pre-Design Investigation (PDI) and Bench Scale Treatability Study Investigation. Data collected during this field investigation and validated for this DUAR are usable as reported with the data validation qualifiers added. Fifteen soil individual sample analyte results (2%) were rejected. A summary of the validation is presented in Section 5. Specific details of the validation are provided in the individual summaries and data validation reports in Appendix A.

Table 1-1
Bench Scale Study Sample and Analysis Summary
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

Sample #	Sample Date	Sample Time	Location	Matrix	Sample Type	Parent Sample #	Analyses
SS-M-1500-051717	5/17/2017	15:00	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-6000-051917	5/19/2017	14:00	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-96000-051917	5/19/2017	14:00	0-18 ft bgs	Soil	Duplicate	SS-H-6000-051917	TAL Metals/Total Hg/Hg Speciation
SS-H	6/9/2017	12:20	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals
SS-H-DUP	6/9/2017	12:05	0-18 ft bgs	Soil	Duplicate	SS-H	TAL Metals
SS-H-CPx-10	6/9/2017	12:45	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-CPx-10-OPTI	6/9/2017	13:00	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-CPx-10-Solidified	6/9/2017	11:00	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-FB-15-OPTI	6/9/2017	13:20	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-FB-15-Solidified	6/9/2017	11:45	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-FB-CaO-10-Solidified	6/9/2017	12:14	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-S-25	6/9/2017	12:30	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-S-5	6/9/2017	12:55	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-M	6/9/2017	12:00	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals
SS-M-CPx-5	6/9/2017	12:50	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-M-CPx-5-OPTI	6/9/2017	13:10	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-M-FB-5	6/9/2017	12:40	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-M-FB-CaO-5	6/9/2017	12:45	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-M-FB-CaO-5-OPTI	6/9/2017	13:30	0-18 ft bgs	Soil	Pilot Study Sample		TAL Metals/Total Hg/Hg Speciation
SS-H-CPx-10-Solidified-2-hours	6/27/2017	15:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-2-hours	6/27/2017	15:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-2-hours	6/27/2017	15:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-2:1	6/28/2017	13:30	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-DUP-2:1	6/28/2017	13:20	0-18 ft bgs	Leachate	Duplicate	SS-H-2:1	SPLP RCRA Metals/SPLP Hg
SS-H-CPx-10-2:1	6/28/2017	12:10	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-CPx-10-OPTI-2:1	6/28/2017	11:30	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-CPx-10-Solidified-2:1	6/28/2017	11:00	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-CPx-10-Solidified-24-hours	6/28/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-OPTI-2:1	6/28/2017	11:50	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-FB-15-Solidified-2:1	6/28/2017	11:10	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-FB-15-Solidified Dup-2:1	6/28/2017	11:11	0-18 ft bgs	Leachate	Duplicate	SS-H-FB-15-Solidified-2:1	SPLP RCRA Metals/SPLP Hg
SS-H-FB-15-Solidified-24-hours	6/28/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-2:1	6/28/2017	11:20	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-FB-CaO-10-Solidified-24-hours	6/28/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-S-25-2:1	6/28/2017	12:30	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-S-5-2:1	6/28/2017	12:40	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-2:1	6/28/2017	13:10	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-CPx-5-2:1	6/28/2017	12:20	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-CPx-5-OPTI-2:1	6/28/2017	11:40	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-FB-5-2:1	6/28/2017	12:50	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-FB-CaO-5-2:1	6/28/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-M-FB-CaO-5-OPTI-2:1	6/28/2017	12:00	0-18 ft bgs	Leachate	Pilot Study Sample		SPLP RCRA Metals/SPLP Hg
SS-H-CPx-10-Solidified-48-hours	6/29/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg

Table 1-1
Bench Scale Study Sample and Analysis Summary
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

Sample #	Sample Date	Sample Time	Location	Matrix	Sample Type	Parent Sample #	Analyses
SS-H-FB-15-Solidified-48-hours	6/29/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-48-hours	6/29/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-72-hours	6/30/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-72-hours	6/30/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-72-hours	6/30/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-8-days	7/5/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-8-days	7/5/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-8-days	7/5/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-14-days	7/11/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-14-day	7/11/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-14-days	7/11/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-21-days	7/18/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-21-day	7/18/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-21-days	7/18/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-28-days	7/25/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-28-day	7/25/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-28-days	7/25/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-CPx-10-Solidified-42-days	8/8/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-15-Solidified-42-day	8/8/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg
SS-H-FB-CaO-10-Solidified-42-days	8/8/2017	13:00	0-18 ft bgs	Leachate	Pilot Study Sample		Total Hg

Notes:

ft bgs = feet below ground surface

Hg = mercury

TAL = target analyte list

SPLP = synthetic precipitation leaching procedure

Section 2

Quality Assurance Objectives

Quality assurance (QA) objectives for data are expressed in terms of measurement performance criteria for the data quality indicators (DQIs). The DQIs are precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). The DQIs provide a mechanism for ongoing QC and for evaluating and measuring data quality throughout the project. QA objectives and DQI measurement performance criteria are outlined in the QAPP.

A review of the generated data is necessary to identify if the measurement performance criteria and objectives established in the QAPP have been met. In general, the following data measurement objectives were evaluated:

- Achievement of analytical method and reporting limit (RL) requirements;
- Adherence to and achievement of appropriate laboratory analytical QC requirements;
- Achievement of the DQIs measurement performance criteria;
- Adherence to sampling and sample handling procedures; and
- Adherence to the sampling design and deviations documented on field change notifications.

Data verification, data validation and data assessment were used to verify adherence to the QAPP procedures and requirements. These assessments were used to reconcile the planned objectives detailed in the QAPP against the investigation results. The outputs serve to verify that the collected data are of sufficient quality to support their intended use.

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Section 3

Summary of Field and Laboratory QA Activities

CDM Smith completed field sampling and preparation and subsampling activities in accordance with the approved work plan and QAPP.

Table 1-1 shows the subsamples prepared and methods analyzed for chemical analyses. All sample identifications were accurately documented by the laboratory and verified by the sampling team.

3.1 Deviations from Field and Laboratory Procedures

No field deviations were noted for these sampling activities. There was a modification for the standard operating procedure (SOP) utilized at the CDM Smith DTL. This modification involved the order of steps to be taken during the sample preparation procedure. Instead of adding mercury to the soil prior to soil compositing and splitting activities, mercury was added to each split sample at the required concentrations after creation of the individual split. Each spiked split sample underwent sample mixing in a rotary tumble. This deviation did not impact project objectives and was approved prior to implementation.

3.2 Field Quality QA/QC

Field QC samples such as matrix spikes/matrix spike duplicates (MS/MSDs) and field duplicates were to be collected at the frequencies (five %) defined in the QAPP. The number of MS/MSDs and field duplicate samples collected satisfies the minimum requirements of one per twenty samples as described in the QAPP.

CDM Smith DTL QA/QC objectives were accomplished through the use of appropriate sampling techniques and collection of the required QC samples at the required frequencies.

3.3 CDM Smith DTL and Subcontractor Laboratory QA/QC

Analytical QA/QC was assessed by CDM Smith DTL and subcontractor laboratory QC checks, method blanks, sample custody tracking, sample preservation, adherence to holding times, laboratory control samples (LCSs), MS/MSDs, sample duplicates, post digestion spikes, calibration verification recoveries, serial dilutions, interference check standards, internal standards, and other applicable QC parameters. The laboratory QC sample results met project requirements with some exceptions as documented in the data validation reports; the appropriate qualifiers were applied to outliers.

3.3.1 Laboratory Methods

Samples were prepared by the CDM Smith DTL, utilizing the following additives and application methods to determine their effectiveness in converting Hg^0 to HgS :

- Use of elemental sulfur and simulation of in situ auger mixing;
- Use of elemental sulfur in a rotary ball mill;
- Use of calcium polysulfide and simulation of in situ auger mixing; and
- Use of FerroBlack® and simulation of in situ auger mixing.

The bench scale study included the steps and procedures detailed in the Final Bench Scale Treatability Study Work Plan (CDM Smith 2017) and the associated SOPs as follows:

- SOP 1-1: Soil Sample Preparation;
- SOP 1-2: Soil Mixing with Additives;
- SOP 1-3: Hg⁰ Analysis in Solid Samples;
- SOP 1-4: SPLP and SDL Procedure on Stabilized Soils; and
- SOP 1-5: Unconfined Compressive Strength (Pocket Penetrometer).

Bench scale study sample results are presented in the Draft Bench Scale Treatability Study Report (CDM Smith 2017).

As stated previously, a subset of the samples prepared and analyzed by the CDM Smith DTL were then sent to the Brooks Applied Laboratory for mercury speciation analysis. EPA CLP laboratories analyzed selected treated soil samples for metals, and CDM Smith DTL created leachates for RCRA metals and total mercury. These samples were then analyzed using the following methods:

Brooks Applied Laboratory

- Mercury - EPA Method 1631;
- Mercury Speciation - 5-Step Extraction (SOP BAL-3900); and
- Percent Total Solid - Standard Method (SM) 2540G.

EPA Laboratories

- Metals and Mercury – Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) ISM02.4.

All the methods are consistent with the QAPP listed methods and provide results that meet the required data quality.

Section 4

Data Validation Procedures

Data were validated by CDM Smith and USEPA Region 2 data validators not associated with the sampling activities. The data validation was performed in accordance with the following documents: specified analytical methods; the project Final QAPP (CDM Smith 2017); EPA National Functional Guidelines for Superfund Inorganic Methods Data Review (January 2017); EPA's Region II validation criteria and SOPs, HW-3A (Rev 1) and HW-3C; and professional judgment.

The following sample delivery group (SDG) data packages were validated:

Brooks Applied Laboratory Data - CDM Smith Validated SDG

- SDG SK5807

EPA Laboratory Data - EPA Region 2 Validated SDGs

- SDG MBDSP4
- SDG MBDWR3
- SDG MBDWS9
- SDG MBDWW6
- SDG MBDWY1
- SDG MBDX59

The bench scale treatability study sample preparation procedures and results were not validated per se, but reviewed for QC parameters such as calibration standards, sand blanks and Hg⁰ versus black and red cinnabar reagent grade HgS.

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Section 5

Data Quality Indicators

The data validation reports were evaluated to determine whether the quality of the data collected achieved the DQOs specified in the QAPP. Data quality and usability were determined based on the results of the DQIs shown in the table below.

DQIs are defined in the following sections. QC parameters evaluated in the data review/validation and the corresponding DQIs are summarized in Table 5-1. All collected data received a Level IV evaluation and the sample preparation procedures were reviewed for industry standard QC practices.

5.1 Precision

Precision is a quantitative term that estimates the reproducibility of a set of replicate measurements under a given set of conditions. It is defined as a measurement of mutual agreement between measurements of the same property and is expressed in terms of RPD between duplicate determinations.

RPD is calculated as follows:

$$\text{RPD} = \text{absolute value } [(C1-C2)/\{(C1+C2)/2\}] \times 100\%$$

Where:

C1 = concentration of primary sample

C2 = concentration of duplicate sample

Field and analytical precision were determined from the review of the field, laboratory duplicates and laboratory spike duplicate (LCSs and MSs) results. The sample results were compared by calculating their RPDs. The field duplicate samples were collected in the same manner as the original samples but were collected in separate, individual containers; given separate sample identifiers; and treated as individual samples by the laboratory.

For field duplicate review, the RPD control limits identified in the QAPP were used. The individual validation reports (Appendix A) and Tables 5-2 and 5-3 identify field duplicate results.

A summary of data qualified based on laboratory or field precision criteria is presented below.

Brooks Applied Laboratory Data - CDM Smith Data Validation Precision Results

- Mercury and mercury speciation – All RPD results are within field duplicate criteria control limits.

- Water soluble mercury, weak-acid soluble mercury, organo-complexed mercury, strongly-bound mercury, mineral-bound mercury and volatile Hg⁰ speciation results were qualified as estimated “J” for all samples based on laboratory duplicate RPDs being outside of criteria.

The laboratory reported the total mercury MS and MSD samples were under-spiked and therefore the MS/MSD results are not valid indicators of data quality. The RPD results for these samples were not able to be evaluated.

The laboratory also reported poor RPD results for six of the samples when comparing the sum of the selective sequential extraction (SSE) fractions to the total mercury results for each sample. The RPD ranged from 33 to 96% for five of the treated spiked soils (positive RPD results from total mercury being greater than the sum of the fractions) and was -55% for SS-M-1500 spiked soil (without treatment). The five-step procedure followed by the laboratory is not designed to provide total values for mercury in the sample but to show how extractable the mercury is from the sample in each fraction. The laboratory noted it is not uncommon for the total values to not perfectly match the sum of the species. No sample analytes were qualified based on these RPD results as applicable qualifiers were applied for the laboratory duplicate criteria.

EPA Laboratory Data - EPA Region 2 Data Validation Precision Results

The following analytes were qualified as estimated for the applicable field duplicate results based on RPD criteria:

- Arsenic, barium, beryllium, chromium, nickel, zinc and mercury.

All laboratory RPDs were within control limits.

No discernable pattern or reason for the exceedances exists. No other field sampling issues were identified from the RPD results that were outside criteria; the exceedances are reasonable for this type of sampling activity.

CDM Smith DTL Data – Bench Scale Treatability Study Data Review

As discussed in the Draft Bench Scale Treatability Study Report (CDM Smith 2017) report, a comparison of the samples prepared and analyzed during the bench scale treatability study at the CDM Smith DTL and the prepared samples sent to Brooks Applied Laboratory and analyzed by SSE method was performed. RPDs were calculated for Hg⁰ and the mineral-bound mercury and mercury sulfide results for the samples analyzed by the two different laboratories.

The Brooks Applied Laboratory speciation results did not exhibit as much conversion of Hg⁰ to mercury sulfide as shown by the CDM Smith DTL method when compared to the Brooks Applied Laboratory method. The Brooks Applied Laboratory results were typically higher in Hg⁰ and lower in mercury sulfide than reported by CDM Smith DTL. However as discussed previously, the Brooks Applied Laboratory method had poor mass balance (total mercury compared to the sum of the extracts) in many samples.

No qualification of data was applied based on the RPDs between the two different laboratory methods due to the nature of the bench scale study being performed and the inherent differences between the methods.

5.2 Accuracy

Accuracy is the degree of agreement of a measurement with an accepted reference or true value and is a measure of the bias in a system. Accuracy of the data was assessed by comparing LCS recovery, MS recovery, calibration recovery, and tracer recoveries with the established criteria. Accuracy is expressed as % recovery (%R), which was calculated by:

$$\text{Percent Recovery} = \frac{(\text{Total Analyte Found} - \text{Amount Original Analyte})}{\text{Amount Analyte Added}} \times 100$$

Analytical accuracy for the entire data collection activity is difficult to measure because several sources of error exist. Errors can be introduced by any of the following:

- Sampling procedure;
- Field contamination;
- Sample preservation and handling;
- Sample matrix;
- Sample preparation; and
- Analytical techniques.

Accuracy is improved by adhering to the approved field and analytical SOPs. The laboratory data were reviewed for accuracy by examining the reported calibrations, MS/MSD recoveries, LCS/LCS duplicate (LCSD) recoveries, serial dilution results, ICP AES results and standard reference material (SRM) as applicable to each analysis.

A summary of data qualified based on QC sample accuracy results is presented below. Appendix A presents the individual data validation reports which specify the qualifications and the samples affected.

Brooks Applied Laboratory Data - CDM Smith Data Validation Accuracy Results

- Mercury speciation – water soluble mercury, weak-acid soluble mercury, strongly-bound mercury, mineral-bound mercury and organo-complexed mercury results were qualified as estimated based on SRM %Rs. Associated sample results were qualified as estimated “J/UJ.”

The laboratory reported that there are no certified SRMs for the SSE procedure commercially available. The SRM used for Hg⁰, HgS, and mercuric chloride (HgCl₂) were produced by Studio Geochemica (SGC) and are not officially certified for any analyte. The laboratory stated that SGC provided expected concentrations for the total mercury and each fraction, and that they have been able to confirm those concentrations during analysis.

In addition, the original soil samples spiked with Hg^0 , SS-M-1500 and SS-H-6000, showed only 26.6% of the mercury in the F4 fraction Hg^0 for SS-M-1500 and only 46.6/48.8% (duplicate samples) of the mercury in the F4 fraction for SS-H-6000. According to the CDM Smith DTL original (before spiking) results for the composite soil samples and the amount of Hg^0 added, these values for Hg^0 (fraction F4) should have been 81% and 96%, respectively for SS-M-1500 and SS-H-6000.

The laboratory stated SRM %Rs were outside the expected results and since no official control limits have been established for the SSE procedure, these outlier recoveries are not indicative of poor data quality. They also noted an observed shift in some recoveries due to SRM degradation. This was noted mostly for the recovery of Hg^0 (expected 1.3%; recovered 13.2%) from HgCl_2 (expected 75.5%; recovered 40.9%) in the SRM shifting from the F1 step to the F2 and F3 steps. The laboratory also noted there is more Hg^0 in the HgS SRM now available in the F3 step. This makes it less available in the F5 step. Historically, NIST2710 mainly recovers in the F5 step. For this set of data the recoveries were as follows: F4 Step: expected 43.0%; recovered 72.8%; and 5F Step: expected 50.5%; recovered 39.6%). The recovery for the F5 step was significantly lower than expected. HgS has been shown to recover mostly in step F5 although it has recovered higher than expected in the F3 fraction (expected 0.0%; recovered 4.2%). Hg^0 mainly recovers in the F4 step although it was higher than expected in steps F1 and F3 which is why the F4 step concentration was lower (expected 89.4%; recovered 72.1%). HgCl_2 usually recovers in step F1 but for these sample results it was recovered more in the F3 step (expected 4.2%; recovered 15.0%). No new SRMs were acquired or used by the laboratory for these samples. As discussed above, CDM Smith qualified results associated with the SRMs outside expected criteria as estimated.

EPA Laboratory Data - EPA Region 2 Data Validation Accuracy Results

Interference Check Standard

- Applicable nondetect selenium results were qualified as rejected based on interference check standard recovery that fell below 50%, which indicates the possibility of false negatives.

Matrix Spikes

- Applicable antimony, arsenic, barium, selenium, silver, and mercury results were qualified as estimated based on MS/MSD criteria.

CDM Smith DTL Data – Bench Scale Treatability Study Data Review

As discussed in the Draft Bench Scale Treatability Study Report (CDM Smith 2017), the bench scale treatability study, individual subsamples (i.e., standards) of Hg^0 , black cinnabar, and red cinnabar were created at concentrations of 1,000 milligram per kilogram in sand. Five individual analyses were performed to assess the homogeneity of the subsamples after spiking and mixing. RSDs were calculated for these analyses. Specific details of these results are in the Draft Bench Scale Treatability Study Report (CDM Smith 2017) report but in general, the CDM Smith DTL method is able to provide reproducible results for total mercury, but more variability is shown in the quantitation of mercury sulfides and possibly more stable mercury species. The soils did not show significant differences in mercury content.

No qualifiers were applied to the bench scale study data results based on RSD results due to the nature of the study.

Sample preservation, handling, and holding times are additional measures of accuracy of the data. All criteria were met for these parameters.

5.2.1 Blank Contamination

Laboratory method blanks are analyzed to identify possible sources of contamination. Contamination of a sample can be introduced by field sample collection methods, sample handling, preparation, and/or analysis. The following sections discuss blank contamination validation actions.

Brooks Applied Laboratory Data - CDM Smith Data Validation Blank Contamination Results

- One sample result for weak-acid soluble mercury was qualified as nondetect based on laboratory blank criteria.

EPA Laboratory Data - EPA Region 2 Data Validation Blank Contamination Results

- Applicable results for the following analytes were qualified as nondetect based on laboratory blank results: arsenic; beryllium; cobalt; selenium; and silver.

CDM Smith DTL Data – Bench Scale Treatability Study Data Review

- Sand blanks were analyzed by the CDM Smith DTL and no measurable concentrations of mercury were detected. No qualification of data was required.

5.3 Representativeness

Representativeness is a qualitative term that expresses the degree to which the sample data accurately represent the environment. The sampling plan and procedures were designed to maximize sample representativeness. Appropriate laboratory QA/QC requirements were described in the QAPP and laboratory statements of work to ensure that the analytical results were representative of the samples collected.

Representativeness can also be monitored by reviewing field documentation and/or by performing field audits. For this report, a detailed review was performed on the chain of custody forms, field data collection forms, and data validation packages.

Field sampling accuracy was attained through strict adherence to the approved QAPP using EPA analytical methods for sample analyses. Based on this, the data should represent as near as possible the actual field conditions at the time of sampling.

Representativeness, as defined above, is believed to have been met. The data collected are suitable for a representative characterization of the sampled areas.

5.4 Comparability

Comparability is the confidence with which one data set can be compared to another data set. Using SMs and units throughout the data generation processes ensures the comparability of data generated in separate sampling days or events. SMs and units were utilized for all sampling

events. All data sets are considered comparable to the degree the bench scale treatability study results and the Brooks Applied Laboratory SSE method results can be compared to each other. Both methods are not standard industry methods so there is inherent variability with results for both methods.

5.5 Completeness

Completeness of the field program is defined as the percentage of samples planned for collection as listed in the final work plan versus the actual number of samples collected during the field program (see equation A).

Completeness for acceptable data is defined as the percentage of acceptable data of known quality obtained and judged to be valid versus the total quantity of data generated (see equation B). Acceptable data include both data that pass all the QC criteria (unqualified data) and data that may not pass all the QC criteria but had appropriate corrective actions taken (qualified but usable data).

$$A. \quad \%Completeness = C \times \frac{100}{n}$$

Where:

C = actual number of samples collected

n = total number of samples planned

$$B. \quad \%Completeness = V \times \frac{100}{n'}$$

Where:

V = number of measurements judged valid

n' = total number of measurements made

All samples outlined in the QAPP were collected as planned or as determined in the field to meet project quality objectives. The completeness for the number of samples planned to be collected versus the number of samples collected was 100% for all analyses.

Fifteen inorganic soil samples results (2%) were rejected. Table 5-4 shows the completeness results for all analyses and media. The 90% completeness goal for usable data has been met for all data.

5.6 Sensitivity

Sensitivity depicts the ability of an analytical system (i.e., sample preparation and instrumental analysis) to detect a target component in a given sample matrix with a defined level of confidence. Factors affecting the sensitivity of an analytical system include: analytical system background (e.g., laboratory artifact or method blank contamination), sample matrix (e.g., co-elution of peaks, or baseline elevation), low level calibration verification standards.

To evaluate if the analytical sensitivity achieved the project expectations, sample-specific quantitation limits (QLs)/RLs were compared against the project action limits and project QL goals set forth in the QAPP. In addition, sample results were compared to detections of target

analytes in method blanks, trip blanks, and equipment rinsate blanks to identify potential effects of laboratory background and field procedures on sensitivity.

Laboratory results are reported according to rules that provide established certainty of detection and RLs. The result for an analyte is flagged with a "U" if that analyte was not detected, or qualified with a "J" flag if associated QC results fall outside the appropriate tolerance limits. Also, if an analyte is present at a concentration between the method detection limit (MDL) and the RL, the analytical result is flagged with a "J," indicating an estimated quantity. Qualifying the result as an estimated concentration reflects increased uncertainty in the reported value.

Qualifiers were applied to applicable sample results by the laboratory and identified during the validation process based on sample results being reported as detected below the MDL/RL.

For the data validated, RLs for most of the sample results were low enough to compare to the RLs in the QAPP. Some sample dilutions were required. RLs above those stated in the QAPP will be evaluated on a case by case basis to determine if project objectives are still met.

Table 5-1 DQIs and Corresponding QC Parameters

Data Quality Indicators	QC Parameters Evaluation in Data Review/Validation
Precision	Relative Percent Difference (RPD) values of: <ol style="list-style-type: none"> 1) Laboratory Control Sample (LCS)/LCS duplicate (LCSD) 2) Matrix Spike (MS)/MS duplicate (MSD) 3) Laboratory and Field duplicates Relative Standard Deviation (RSD) values of: <ol style="list-style-type: none"> 1) Initial calibration verifications 2) Initial precision and recovery standards
Accuracy/Bias	Percent Recovery (%R) or Percent Difference (%D) values of: <ol style="list-style-type: none"> 1) Initial calibration and continuing calibration verification 2) LCS/LCSD 3) MS/MSD 4) Serial dilution (ICP metals) 5) ICP inter-element interference check samples Results of: <ol style="list-style-type: none"> 1) Instrument and calibration blanks 2) Method (preparation) blanks 3) Temperature blanks
Representativeness	Results of all blanks Sample integrity (Chain-of Custody and sample receipt forms) Holding times Compound identification (retention times, mass spectra)
Comparability	Sample-specific reporting limits (RLs) Sample collection methods and laboratory analytical methods
Completeness	Laboratory deliverables Data qualifiers and Requested/reported valid results Field sample collection (primary and QC samples) Contract compliance (i.e., method and instrument QC within limits)
Sensitivity	Method RLs Adequacy of sample dilution

Table 5-2
Bench Scale Study Soil Sample Duplicate Relative Percent Difference Results
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

Location			Bench Scale Area - H		Bench Scale Area - H		RPD	ABS	Bench Scale Area - H		Bench Scale Area - H		RPD	ABS
Sample #			SS-H		SS-H-DUP				SS-H-6000-051917		SS-H-96000-051917			
Sample Date			6/9/2017		6/9/2017				5/19/2017		5/19/2017			
Inorganics (mg/kg)		CRQL												
Aluminum	20	--	5150		4700		9.1	NA	8270		8300		0.4	NA
Antimony	6	--	1.2	J	1.2	J	0.0	0.00	1.7	J	1.2	J	34.5	0.50
Arsenic	1	--	245		209		15.9	NA	281		217	J	25.7	NA
Barium	20	--	588		526		11.1	NA	971		1030	J	5.9	NA
Beryllium	0.5	--	1.3	J	0.36	J	113.3	0.94	0.55	U	0.55	U	NC	NC
Cadmium	0.5	--	5.1		3.7		31.8	NA	3.7		3.5		5.6	NA
Calcium	500	--	33400		31800		4.9	NA	38700		40000		3.3	NA
Chromium	1	--	48.2		42.2		13.3	NA	57.7		56.8		1.6	NA
Cobalt	5	--	3.9	J	3.3	J	16.7	0.60	5.8		5.5	U	NC	0.30
Copper	2.5	--	130		78.7		49.2	NA	99.1		101		1.9	NA
Iron	10	--	14300		13000		9.5	NA	16200		18500		13.3	NA
Lead	1	--	110		105		4.7	NA	124		122		1.6	NA
Magnesium	500	--	3640		3380		7.4	NA	4400		4360		0.9	NA
Manganese	1.5	--	124		110		12.0	NA	143		142		0.7	NA
Nickel	4	--	9.4		7.4		23.8	2.00	12	J	23	J	62.9	11.00
Potassium	500	--	635		597		6.2	38.00	1420		1430		0.7	10.00
Selenium	3.5	--	2.8	R	2.8	R	NC	NC	3.8	U	3.8	U	NC	NC
Silver	1	--	0.8	U	0.79	U	NC	NC	1.1	U	1.1	U	NC	NC
Sodium	500	--	1560		1390		11.5	170.00	1710		1680		1.8	30.00
Thallium	2.5	--	2	U	2	U	NC	NC	2.7	U	2.7	U	NC	NC
Vanadium	5	--	17.7		16		10.1	1.70	26		25.1		3.5	NA
Zinc	6	--	362	J	189	J	62.8	NA	376		318		16.7	NA

Table 5-2
Bench Scale Study Soil Sample Duplicate Relative Percent Difference (RPD) Results
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

Location				Bench Scale Area - H		Bench Scale Area - H		RPD	ABS	Bench Scale Area - H		Bench Scale Area - H		RPD	ABS	
Sample #				SS-H		SS-H-DUP				SS-H-6000-051917		SS-H-96000-051917				
Sample Date				6/9/2017		6/9/2017				5/19/2017		5/19/2017				
Total Mercury (ng/g)																
Mercury			0.10	--	NS		NS		NC	NA	6990000	J	6910000	J	1.2	NA
Mercury Speciation (ng/g)																
Volatile Hg			NA	F0	NS		NS		NC	NA	17300	J	21400	J	21.2	NA
Water-soluble Hg			NA	F1	NS		NS		NC	NA	23700	J	30900	J	26.4	NA
Weak acid-soluble Hg			NA	F2	NS		NS		NC	NA	280000	J	335000	J	17.9	NA
Organo-complexed Hg			NA	F3	NS		NS		NC	NA	405000	J	493000	J	19.6	NA
Strongly-complexed and elemental Hg			NA	F4	NS		NS		NC	NA	2940000	J	3820000	J	26.0	NA
Mineral-bound Hg			NA	F5	NS		NS		NC	NA	2610000	J	3130000	J	18.1	NA
Total Solids			NA	--	NS		NS		NC	NA	92.01	J	92.34	J	0.4	NA

Notes:

% = percent

ABS = absolute difference

NA = not applicable

mg/kg = milligrams per kilogram

ng/g = nanograms per gram

NS = not sample

NC = not calculable

RPD = relative percent difference

CRQL = contract required quantitation limit

Data Validation Qualifiers:

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

RPD criteria:

Metals : 100%

Total Mercury: 100%

Abs. Criteria: Either sample result is less than 5 times the CRQL and the absolute difference between the results < 2xs the CRQL

Bold/Highlighted Cell: RPD result is greater than the RPD criteria or the absolute difference exceeds the absolute difference criteria

Table 5-3

Bench Scale Study Water Leachate Sample Duplicate Relative Percent Difference Results

LCP Chemicals, Inc. Superfund Site

Linden, New Jersey

Location		Bench Scale Area - H		Bench Scale Area - H		RPD	ABS	Bench Scale Area - H		Bench Scale Area - H		RPD	ABS
Sample #		SS-H-2:1		SS-H-DUP-2:1				SS-H-FB-15-SOLIDIFIED-2:1		SS-H-FB-15-SOLIDIFIED DUP-2:1			
Sample Date		6/28/2017		6/28/2017				6/28/2017		6/28/2017			
Inorganics (ug/L)	CRQL												
Arsenic	10	232	J	180	J	25.2	NA	10	U	13.9		NC	3.90
Barium	200	358	J	20.9	J	177.9	337.10	84.5	J	91.4	J	7.8	6.90
Cadmium	5	1.9	J	1	J	62.1	0.90	5	U	5	U	NC	NC
Chromium	10	15.6	J	3.5	J	126.7	12.10	34.2		35.5		3.7	1.30
Lead	10	18.9		10	U	NC	8.90	10	U	10	U	NC	NC
Mercury	0.2	408	J	28.7	J	173.7	NA	13700		13200		3.7	NA
Selenium	35	35	U	3.9	J	NC	31.10	14.2	J	14.6	J	2.8	0.40
Silver	10	10	U	10	U	NC	NC	10	U	10	U	NC	NC

Notes:

% = percent

ABS = absolute difference

NA = not applicable

NS = not sample

NC = not calculable

RPD = relative percent difference

ug/L = micrograms per liter

CRQL = contract required quantitation limit

Data Validation Qualifiers:

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

RPD criteria:

Metals : 50%

Total Mercury: 50%

Abs. Criteria: Either sample result is less than 5 times the CRQL and the absolute difference between the results < 2xs the CRQL**Bold/Highlighted Cell:** RPD result is greater than the RPD criteria or the absolute difference exceeds the absolute difference criteria

Table 5-4
Bench Scale Study Sampling Completeness
LCP Chemicals, Inc. Superfund Site
Linden, New Jersey

Matrix	Method	No Qualifier	J	J+	R	U	UJ	Percent Estimated	Percent Rejected	Percent Non-Detect	Percent Detected
Soil	BAL3900		89				1	99%	0%	1%	99%
Soil	E160.3		15					100%	0%	0%	100%
Soil	E1631E		15					100%	0%	0%	100%
Soil	E200.7	288	32		15	38	1	9%	4%	10%	86%
Water Leachate	E200.7	26	34			44	1	32%	0%	43%	57%
Water Leachate	E245.2	40	1	1				5%	0%	0%	100%
TOTAL SOIL:		288	151	0	15	38	2	31%	3%	8%	89%
TOTAL LEACHATE:		66	35	1	0	44	1	24%	0%	31%	69%
TOTAL:		354	186	1	15	82	3	29%	2%	13%	84%
PERCENT COMPLETE:	98%										

Data Validation Qualifiers:

J = The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.

J+ = The result is an estimated quantity, but the result may be biased high.

R = The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

UJ = The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

Section 6

Assessment of Data Usability and Reconciliation with Work Plan Goals

The CDM Smith bench scale treatability study data are usable as reported. The data results are considered defensible based on method and laboratory procedures and are usable for project decisions. Precision and accuracy of the bench scale procedures was demonstrated by RPDs and RSDs that were within standard criteria and sand blanks that had minimal detected concentrations of mercury.

The Brooks Applied Laboratory confirmation sample results were all qualified as estimated for various QC parameters. The laboratory also reported in their case narrative that quantified results for both samples and the SRMs “can be somewhat variable and therefore the method is not completely effective for determining the concentrations of individual fractions in each sample.” The laboratory reported that the method has been shown to be “effective as a qualitative assessment of the relative percentages of each mercury fraction.” Based on professional judgement and past experience with this laboratory for this same method, the confirmation sample results are considered to be useful for a general comparison of the sample results compared to the treatability study results. However, the treatability study results from the CDM Smith DTL are considered more reliable and defensible due to the stringent method procedures performed and followed for sample analyses. The Brooks Applied Laboratory data case narrative also stated the SRMs used during analyses had signs of degradation and no new SRMs were used during the analyses of the samples, indicating the sample results are definitely estimated concentrations and should be used with a degree of caution.

The bench scale study samples prepared by the CDM Smith DTL and analyzed by EPA laboratories had acceptable results that are considered usable with the appropriate qualifiers applied except for the 15 soil selenium results that were rejected. The rejected sample results are not usable for project decisions.

Over 90% of the data reported and validated in this report are suitable for their intended use in the PDI report and the treatability study as stated in the QAPP. All planned samples were collected. The DQIs identified in the QAPP mainly met appropriate measurement performance criteria. All data are usable for project decisions with the appropriate qualifiers applied except for the results that were rejected.

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Appendix A

Data Validation Reports

LCP Chemicals, Inc. Superfund Site
Linden, New Jersey
Mercury / Mercury Speciation Data Validation Report

Sample Delivery Group (SDG) Number:
Laboratory:

SK5807
Katahdin Analytical Services

Matrix:
Collection date:
Analysis/Methods:

Soil
5/17/17 5/19/2017 6/9/17

Mercury - USEPA Method 1631
Mercury Speciation - 5-Step Extraction (SOP BAL-3900)
Percent Total Solids - SM 2540G

Samples in SDG:

<u>Sample Number</u>	<u>Sub Lab ID</u>	<u>Sample Number</u>	<u>Sub Lab ID</u>
SS-H-6000-051917	SK5807-1	SS-H-FB-CAO-10-SOLIDIFIED	SK5807-9
SS-H-96000-051917	SK5807-2	SS-H-S-25	SK5807-10
SS-M-1500-051717	SK5807-3	SS-H-S-5	SK5807-11
SS-H-CPX-10	SK5807-4	SS-MCPX-5	SK5807-12
SS-H-CPX-10-OPTI	SK5807-5	SS-MCPX-5-OPTI	SK5807-13
SS-H-CPX-10-OPTI-SOILDIFIED	SK5807-6	SS-M-FB-5	SK5807-14
SS-H-FB-15-OPTI	SK5807-7	SS-M-FB-CAO-5	SK5807-15
SS-FB-15-SOLIDIFIED	SK5807-8	SS-M-FB-CAO-5-OPTI	SK5807-16

Data validation was performed in accordance with the specific analytical methods and the National Functional Guidelines for Inorganic Superfund Methods Data Review (USEPA 2017).

Total Mercury and Mercury Speciation

Precision:	Yes	No	N/A
Are the Total Mercury field duplicate relative percent differences (RPD) $\leq 100\%$ for soils or within CRQL criteria?	Yes		
Are the Methyl Mercury field duplicate relative percent differences (RPD) $\leq 40\%$ for soils or within CRQL criteria?	N/A		
Are the Mercury Speciation field duplicate relative percent differences (RPD) $\leq 35\%$ for soils or within CRQL criteria?	Yes		
Are the Total Mercury matrix spike duplicates RPD $\leq 35\%$?	N/A		
Are the Methyl Mercury matrix spike duplicates RPD $\leq 35\%$?	N/A		
Are the Mercury Speciation matrix spike duplicates RPD $\leq 35\%$?	Yes		
Are the laboratory control sample duplicates RPD $\leq 20\%$?	Yes		
Are the laboratory duplicate RPDs $\leq 35\%$ for soils or within CRQL criteria?	No		
<u>Comments (note deviations):</u>			

<u>Field Duplicates</u>	<u>Sample</u>	<u>Duplicate</u>	<u>MRL</u>	<u>%RPDs</u>	<u>Qualifier</u>	<u>Associated Samples</u>
	SS-H-6000-051917	SS-H-96000-051917		Acceptable		

<u>MS/MSD</u>	<u>Analyte</u>	<u>%R</u>	<u>Limits</u>	<u>RPD %</u>	<u>Qualifiers</u>	<u>Associated Samples</u>
B171654-MS2 / MSD2	Total Mercury	NR				Case narrative states the MS/MSD associated with this batch were under spiked and therefore not valid indicators of data quality; further post digestion spike met QC criteria.

<u>LCSD</u>	<u>Analyte</u>	<u>%R</u>	<u>Limits</u>	<u>RPD %</u>	<u>Qualifiers</u>	<u>Associated Samples</u>
	Not Applicable					

Laboratory Duplicate	Sample	Duplicate	MRL	%RPDs	Qualifier	Associated Samples
Total Hg				Acceptable		
B171656-DUP3 Water Soluble Mercury	12910	5341		83%	J/UJ	All samples
B171657-DUP5 Weak Acid Soluble Mercury	5657	288.7		181%	J/UJ	All samples
B171657-DUP4 Weak Acid Soluble Mercury	23180	808.8		187%	J/UJ	All samples
B171658-DUP3 Organo-complexed Mercury	20310	5724		112%	J/UJ	All samples
B171658-DUP4 Organo-complexed Mercury	9611	23250		83%	J/UJ	All samples
B171659-DUP3 Strongly-Bound Mercury	1084000	506800		73%	J/UJ	All samples
B171660-DUP3 Mineral-Bound Mercury	2939000	1279000		79%	J/UJ	All samples
B171685-DUP1 Volatile Elemental Mercury	7583	863.1		159%	J/UJ	All samples

Accuracy:	Yes	No	N/A
Were serial dilutions analyzed and within control limits of ±10% for waters (± for 15% for soils) or initial sample result less than 50x MDL?			N/A
Was the Total Mercury matrix spike criteria met (frequency 20% and % recovery 75-125%)?			N/A
Was the Methyl Mercury matrix spike criteria met (frequency 20% and % recovery 65-135%)?			N/A
Was the Mercury Speciation matrix spike criteria met (frequency 20% and % recovery 77-123%)?			Yes
Was post digestion spike criteria met (if applicable)?			Yes
Was laboratory control sample criteria met?			Yes
Was laboratory blank criteria met (within control limits)?			No
Were ICV (85-115%)/CCV (77-123%) recoveries within criteria for Total Mercury?			Yes
Were ICV (80-120%)/CCV (67-133%) recoveries within criteria for Methyl Mercury?			N/A
Were ICV (85-115%)/CCV (66-123%) recoveries within criteria for Mercury Speciation?			Yes
Were the Detection Limit PQL Standards within 70-130?			Yes
Was the %D on form 16-IN for the initial calibration instrument response and concentration data <30%?			N/A
Were ICSA/ICSAB % recoveries acceptable or within CRQL criteria?			N/A
Was the tune %RSD <5% ?			N/A
Was internal standard criteria met?			N/A
<u>Comments (note deviations):</u>			

Serial Dilution	Analyte	Initial Sample Result	%D	50 x MDL	Qualifier	Associated Samples
	Not Applicable					
MS	Analyte	%R	Limits	Digestion % R	Qualifier	Associated Samples
B171654-MS2 / MSD2	Total Mercury	NR				Case narrative states the MS/MSD associated with this batch were under spiked and therefore not valid indicators of data quality; further post digestion spike met QC criteria.

SRM	Analyte	%R	Limits	Qualifier	Associated Samples
Total Mercury SRMs					
B171656-SRM2	Water Soluble Mercury	71%	67-133	J/UJ	All samples
B171656-SRM3	Water Soluble Mercury	982%	67-133	J/UJ	All samples
B171656-SRM4	Water Soluble Mercury	53%	67-133	J/UJ	All samples
B171657-SRM1	Weak Acid Soluble Mercury	314%	67-133	J/UJ	All samples
B171657-SRM3	Weak Acid Soluble Mercury	132%	67-133	J/UJ	All samples
B171657-SRM35	Weak Acid Soluble Mercury	155%	67-133	J/UJ	All samples
B171658-SRM3	Organo-complexed Mercury	147%	67-133	J/UJ	All samples
B171658-SRM5	Organo-complexed Mercury	60000%	67-133	J/UJ	All samples
B171658-SRM6	Organo-complexed Mercury	353%	67-133	J/UJ	All samples
B171659-SRM1	Strongly-Bound Mercury	178%	67-133	J/UJ	All samples
B171659-SRM2	Strongly-Bound Mercury	439%	67-133	J/UJ	All samples
B171659-SRM4	Strongly-Bound Mercury	156%	67-133	J/UJ	All samples
B171660-SRM1	Mineral-Bound Mercury	69%	67-133	J/UJ	All samples
B171660-SRM3	Mineral-Bound Mercury	187%	67-133	J/UJ	All samples
B171660-SRM4	Mineral-Bound Mercury	147%	67-133	J/UJ	All samples

ICV/CCV	Analyte	%R	Limits	Qualifier	Associated Samples
Acceptable					

Blanks	Note: ICBs and Prep blanks are associated with all samples (unless otherwise indicated). Individual CCBs are associated with specific samples.				
Prep Blanks	Analyte	Result (ng/g)	MDL/MRL	Qualifiers	Associated Samples
	Weak Acid Soluble Mercury	0.36 / 0.12 / 0.19	111 / 278	MRL U	1721026-04
Numerous analytes were detected in the remainder of the method blanks; however, no qualification was required as associated sample results were > MRL.					

ICB / CCBs	Analyte	Result (ug/L)	MDL/MRL	Qualifier	Associated Samples
Not Applicable					

Field Blanks	Result (ng/L)	MDL/MRL	Qualifier	Associated Samples
Not Applicable				

ICSA/AB	Analyte - Solution A	%R	Found Sol. A / True A	LOD	Qualifier	Associated Samples
Not Applicable						

PQL Standards	Analyte	%R	Limits	Qualifier	Associated Samples
Not Applicable					

Tune	Analyte	%RSD	Limits	Qualifier	Associated Samples
Not Applicable					

Internal Standard	Analyte	%RI	Limits	Qualifier	Associated Samples
Not Applicable					

Initial Calibration %D (Form 16)	Analyte	%D	Limits	Qualifier	Associated Samples
Not Applicable					

Representativeness:		Yes	No	N/A
Were sampling procedures and design criteria met?			Yes	
Were holding times met?			Yes	
Were preservation criteria met? (0 ± 4°C for mercury speciation - 0 ± 6°C for total mercury and methyl mercury)			Yes	
Were Chain-of-Custody records complete and provided in data package?			Yes	
Was the raw data present for drying logs, preparation logs, analytical instrument real-time printouts and laboratory bench sheets?			Yes	
Were results less than MDL reported with a "U" and values less than the LOQs but greater than MDL reported with a "J"?			Yes	
<u>Comments (note deviations):</u> Cooler temperatures were 1.0 & 2.9 degrees C.				

Holding Times	<u>Days to Analysis</u>	<u>HT Criteria</u>	<u>Qualifier</u>	<u>Associated Samples</u>
Acceptable				

Completeness (90%):		Yes	No	N/A
Are all data in this SDG usable?			Yes	
<u>Comments (note deviations):</u>				

Sensitivity:		Yes	No	N/A
Are MDLs present and reported?			Yes	
Do the reporting limits meet the project requirements?			Yes	
<u>Comments (note deviations):</u>				

Overall Comments:
Data is usable as reported with appropriate qualifiers applied.

Data Validator:	<u><i>Kristine Molloy</i></u>	Date:	<u><i>8/24/2017</i></u>
Data Reviewer:	<u><i>Cherie Zakowski</i></u>	Date:	<u><i>8/26/2017</i></u>



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
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2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No.: 46887

Site: LCP Chemicals Inc

Number of Samples: 3 (Soil)

Analysis: Metals (ICP-AES)

SDG No.: MBDSP4

Laboratory: Bonner Analytical Testing Co.

Sampling dates: 5/17/17 – 5/19/17

Validation SOP: HW-3a (Rev 1)

QAPP

Contractor: CDM

Reference: Contract #: W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions.
Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

None

Major Findings:

Samples MBDSP4, MBDSP5 and MBDSP6 have analytes that have been qualified J, J+ or J-.

Minor Findings:

None

COMMENT: Concentrations of soil **Arsenic, Barium, Cadmium, Copper, Lead, Nickel, and Zinc** exceeded the project action levels for one or more samples.

Reviewer Name(s): Jianwei Huang

Approver's Signature:

Date: 07/12/2017

Name: Narendra Kumar

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
U	The analyte was analyzed for, but was not detected above the level of the reported quantitation limit.	The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method	The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5).
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL).
J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: METALS ICP-AES

The current SOP HW-3a (Rev 1) September 2016, USEPA Region II for the evaluation of ICP-AES metals generated through Statement of Work ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi-Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time or pH (aqueous samples are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (180 days) or pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995 . The percent differences calculated for all of the non-zero standards must be within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90 – 110%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

3. BLANK CONTAMINATION



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Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. The preparation blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

The following samples have analyte results less than or equal to CRQLs. The associated CCB analyte results are less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

Beryllium MBDSP4, MBDSP5, MBDSP6

Cobalt MBDSP6, MBDSP4

Selenium MBDSP6, MBDSP5

Silver MBDSP4, MBDSP5, MBDSP6

The following samples have analyte results greater than or equal to MDLs and less than or equal to CRQLs. The associated ICB analyte results are greater than or equal to MDLs and less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

Cobalt MBDSP4, MBDSP6

Silver MBDSP6, MBDSP4, MBDSP5

4. INTERFERENCE CHECK SAMPLE

The Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of ICS Solution must fall within the control limits of $\pm 20\%$ or $\pm \text{CRQL}$ (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are $\geq \text{MDL}$ are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is $\geq \text{MDL}$, the possibility of false negatives in the samples exists. In general, ICP sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

5. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4\times$ the spike added. For a matrix



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spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The following sample is associated with Matrix Spike sample that has spike analyte %R less than 30% and Post-digestion spike analyte %R greater than or equal to 75%. Detects are qualified as J. Non-detects are qualified as UJ.

Antimony MBDSP4

The following sample is associated with Matrix Spike sample that has spike analyte %R within 30 - 74% and Post-digestion spike analyte %R greater than or equal to 75%. Detects are qualified as J. Non-detects are qualified as UJ.

Arsenic and Barium MBDSP4

6. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 35% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

7. FIELD DUPLICATE (MBDSP5/MBDSP6)

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of 2x the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

The following Duplicate and/or original soil sample results are less than 5x the CRQL and the absolute difference between duplicate and original samples is greater than 2x the CRQL. Detected analytes are qualified J. Non-detected analytes are qualified UJ.

Nickel MBDSP5, MBDSP6

8. LABORATORY CONTROL SAMPLE

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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9. ICP SERIAL DILUTION

The serial dilution of samples quantitated by Inductively Coupled Plasma determines whether or not significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 15. For a serial dilution analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the serial dilution sample.

No problems were found for this criterion.

10. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. All results of a sample with percent solids less than 50% are qualified estimated, "J". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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EXECUTIVE NARRATIVE

Case No.: 46975

Site: LCP Chemicals Inc.

Number of Samples: 16 (Soil)

Analysis: Metals (ICP-AES)

SDG No.: MBDWR3

Laboratory: Chemtech Consulting Group

Sampling dates: 06/09/17

Validation SOP: HW-3a (Rev 1)

QAPP

Contractor: CDM

Reference: Contract # W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Samples MBDWR3, MBDWR4, MBDWR5, MBDWR6, MBDWR7, MBDWR8, MBDWR9, MBDWS0, MBDWS1, MBDWS2, MBDWS3, MBDWS4, MBDWS5, MBDWS6, MBDWS7, and MBDWS8 have been qualified R for Selenium due to ICSAB has recovery below 50% and all the samples have interfering analyte (Iron) results greater than the ICS concentration.

Major Findings:

Samples MBDWR3, MBDWS7 and MBDWS8 have analytes that have been qualified J, J+ or J-.

Minor Findings:

None

COMMENT: Concentrations of **Lead, Arsenic, Barium, Cadmium, Copper and Zinc** exceeded the project action levels for one or more samples.

Reviewer Name(s): Dharmesh Patel

Approver's Signature:

Date: 08/17/17

Name: Russell Arnone

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
U	The analyte was analyzed for, but was not detected above the level of the reported quantitation limit.	The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method	The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5).
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL).
J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: METALS ICP-AES

The current SOP HW-3a (Rev 1) September 2016, USEPA Region II for the evaluation of ICP-AES metals generated through Statement of Work ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi-Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time or pH (aqueous samples are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (180 days) or pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995 . The percent differences calculated for all of the non-zero standards must be within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s).

The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90 – 110%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. The preparation blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

The following samples have analyte results greater than or equal to MDLs and less than or equal to CRQLs. The associated ICB analyte results are greater than or equal to MDLs and less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

Silver MBDWR4, MBDWR6, MBDWS0, MBDWS2, MBDWS3, MBDWS7.

4. INTERFERENCE CHECK SAMPLE

The Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of ICS Solution must fall within the control limits of $\pm 20\%$ or $\pm \text{CRQL}$ (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are $\geq \text{MDL}$ are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is $\geq \text{MDL}$, the possibility of false negatives in the samples exists. In general, ICP sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

The ICSAB recovery for an analyte falls below 50%, therefore the possibility of false negative exists. The following associated non-detected samples are qualified as unusable (R).

Selenium MBDWR3, MBDWR4, MBDWR5, MBDWR6, MBDWR7, MBDWR8, MBDWR9, MBDWS0, MBDWS1, MBDWS2, MBDWS3, MBDWS4, MBDWS5, MBDWS6, MBDWS7, MBDWS8.

5. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The following sample has matrix spike percent recovery in the range of 30 – 74% and Post-digestion spike sample has percent recovery greater than or equal to 75%. Detects are qualified as J. Nondetects are qualified as UJ.

Selenium MBDWR3.



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The following sample has matrix spike percent recovery in the range of 30 – 74% and the post digestion spike is not required. Detected analyte with result greater than or equal to MDL is qualified J. Non-detected analytes are qualified UJ.

Silver MBDWR3

6. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 35% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

7. FIELD DUPLICATE: MBDWS8/MBDWS7

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the 2xCRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.

The following Duplicate and original soil sample results are greater than or equal to 5x the CRQL and RPD is greater than 50%. Detected analytes are qualified J.

Zinc MBDWS8, MBDWS7.

The following Duplicate and/or original soil sample results are less than 5x the CRQL and absolute difference between duplicate and original samples are greater than 2x the CRQL. Detected analytes with results greater than MDL are qualified J. Non-detected analytes are qualified UJ.

Beryllium MBDWS8, MBDWS7.

8. LABORATORY CONTROL SAMPLE

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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9. ICP SERIAL DILUTION

The serial dilution of samples quantitated by Inductively Coupled Plasma determines whether or not significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 15. For a serial dilution analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the serial dilution sample.

No problems were found for this criterion.

10. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. All results of a sample with percent solids less than 50% are qualified estimated, "J". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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EXECUTIVE NARRATIVE

Case No.: 46975

Site: LCP Chemicals Inc.

Number of Samples: 17 (Aqueous)

Analysis: Metals (ICP-AES) and Mercury

SDG No.: MBDWS9

Laboratory: Chemtech Consulting Group

Sampling dates: 06/28/17

Validation SOP: HW-3a, and -3c (Rev 1)

QAPP

Contractor: CDM

Reference: Contract # W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

None

Major Findings:

Samples MBDWS9, MBDWW4 and MBDWW5 have analytes that have been qualified J, J+ or J-.

Minor Findings:

None

COMMENT: Concentrations of **Lead, Arsenic, Cadmium and Mercury** exceeded the project action levels for one or more samples.

Reviewer Name(s): Dharmesh Patel

Approver's Signature:

Date: 08/17/17

Name: Russell Arnone

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
U	The analyte was analyzed for, but was not detected above the level of the reported quantitation limit.	The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method	The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5).
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL).
J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: METALS ICP-AES

The current SOP HW-3a (Rev 1) September 2016, USEPA Region II for the evaluation of ICP-AES metals generated through Statement of Work ISOM02.2, and any future editorial revisions of ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time or pH (aqueous samples are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (180 days) or pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995 . The percent differences calculated for all of the non-zero standards must be within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90 – 110%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. The preparation blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

The following samples have analyte results greater than or equal to MDLs and less than or equal to CRQLs. The associated ICB analyte results are greater than or equal to MDLs and less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

Silver MBDWT4.

The following samples have analyte results less than or equal to CRQLs. The associated CCB analyte results are less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

Arsenic MBDWT0.

4. INTERFERENCE CHECK SAMPLE

The Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of ICS Solution must fall within the control limits of $\pm 20\%$ or $\pm \text{CRQL}$ (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are $\geq \text{MDL}$ are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is $\geq \text{MDL}$, the possibility of false negatives in the samples exists. In general, ICP sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

5. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The following sample has matrix spike percent recovery in the range of 30 – 74% and the post digestion spike is not required. Detected analyte with result greater than or equal to MDL is qualified J. Non-detected analytes are qualified UJ.



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Silver MBDWS9

6. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

7. FIELD DUPLICATE: MBDWT0/MBDWT1, MBDWW5/MBDWW4

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

The following Duplicate and original water sample results are greater than or equal to 5x the CRQL and RPD is greater than 20%. Detected analytes are qualified J.

Arsenic MBDWW5, MBDWW4.

The following Duplicate and/or original water sample results are less than 5x the CRQL and absolute difference between duplicate and original samples are greater than the CRQL. Detected analytes with results greater than MDL are qualified J. Non-detected analytes are qualified UJ.

Barium MBDWW5, MBDWW4.

Chromium MBDWW5, MBDWW4.

8. LABORATORY CONTROL SAMPLE

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

9. ICP SERIAL DILUTION

The serial dilution of samples quantitated by Inductively Coupled Plasma determines whether or not significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution



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analysis (a five-fold dilution) after correction for dilution shall be less than 10. For a serial dilution analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the serial dilution sample.

No problems were found for this criterion.

10. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. All results of a sample with percent solids less than 50% are qualified estimated, "J". Qualifications were applied to the samples and analytes as shown below.

Not applicable.

ANALYSIS: MERCURY

The current SOP HW-3c (Rev 1) September 2016, USEPA Region II for the evaluation of Mercury generated through Statement of Work ISOM02.2, and any future editorial revisions of ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time, pH (aqueous samples), or cooler temperature are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (28 days) and pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects (sample quantitation limits) will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range. Qualifications were applied to the samples and analytes as shown below.



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No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 85 – 115%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

4. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

No problems were found for this criterion.

5. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

6. FIELD DUPLICATE: MBDWT0/MBDWT1, MBDWW5/MBBWW4

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate



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value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.

The following Duplicate and original water sample results are greater than or equal to $5x$ the CRQL and RPD is greater than 20%. Detected analytes are qualified J.

Mercury MBDWW5, MBDWW4.

7. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. Qualifications were applied to the samples and analytes as shown below.

Not applicable.



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2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No.: 46975

Site: LCP Chemicals Inc.

Number of Samples: 15 (Water)

Analysis: Hg

SDG No.: MBDWW6

Laboratory: Chemtech Consulting Group

Sampling dates: 6/27/17-7/5/17

Validation SOP: HW -3c (Rev. 1)

QAPP

Contractor: CDM Smith

Reference: Contract # W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

None

Major Findings:

None

Minor Findings:

None

COMMENT: The concentration of Hg exceeded the project action level in one or more samples.

Reviewer Name(s): Israel Okwuonu

Approver's Signature:

Date: 08/14/17

Name: Russell Arnone

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
U	The analyte was analyzed for, but was not detected above the level of the reported quantitation limit.	The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method	The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5).
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL).
J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: MERCURY

The current SOP HW-3c (Revision 1) September, 2016 USEPA Region II for the evaluation of Mercury generated through Statement of Work ISOM02.2, and any future editorial revisions of ISOM02.2, has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time, pH (aqueous samples), or cooler temperature are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (28 days) and pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects (sample quantitation limits) will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 85 – 115%. Qualifications were applied to the samples and analytes as shown below.



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No problems were found for this criterion.

3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

4. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

No problems were found for this criterion.

5. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

6. FIELD DUPLICATE

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.

Not applicable.

7. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. Qualifications were applied to the samples and analytes as shown below.

Not applicable.



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EXECUTIVE NARRATIVE

Case No.: 46975
Site: LCP Chemicals Inc.
Number of Samples: 9 (Aqueous)
Analysis: Mercury

SDG No.: MBDWY1
Laboratory: Chemtech Consulting Group
Sampling dates: 07/11/17 to 07/25/17
Validation SOP: HW-3c (Rev 1)

QAPP

Contractor: CDM
Reference: Contract # W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

None

Major Findings:

None

Minor Findings:

None

COMMENT: Concentrations of **Mercury** exceeded the project action levels for one or more samples.

Reviewer Name(s): Dharmesh Patel

Approver's Signature:

Date: 08/25/17

Name: Russell Arnone

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
U	The analyte was analyzed for, but was not detected above the level of the reported quantitation limit.	The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method	The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5).
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL).
J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: MERCURY

The current SOP HW-3c (Rev 1) September 2016, USEPA Region II for the evaluation of Mercury generated through Statement of Work ISOM02.2, and any future editorial revisions of ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time, pH (aqueous samples), or cooler temperature are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (28 days) and pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects (sample quantitation limits) will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 85 – 115%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.



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3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

4. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

No problems were found for this criterion.

5. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

6. FIELD DUPLICATE:

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

Not applicable.

7. PERCENT SOLIDS

The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. Qualifications were applied to the samples and analytes as shown below.

Not applicable.



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EXECUTIVE NARRATIVE

Case No.: 46975

Site: LCP Chemicals Inc.

Number of Samples: 3 (Water)

Analysis: Mercury

SDG No.: MBDX59

Laboratory: Chemtech Consulting Group

Sampling Dates: 8/8/2017

Validation SOP: HW-3c (Rev 1)

QAPP

Contractor: CDM Smith

Reference: Contract #: W912DQ-15-D-3013

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

None

Major Findings:

Sample MBDX61 has analytes that have been qualified J, J+ or J-.

Minor Findings:

None

COMMENT: **Mercury** concentration exceeded the project action level for all samples.

Reviewer Name(s): Jianwei Huang

Approver's Signature:

Date: 09/19/17

Name: Russell Arnone

Affiliation: USEPA/R2/HWSB/HWSS



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Data Qualifier Definitions (National Functional Guidelines)			
Qualifier Symbol	Explanation		
	INORGANICS	ORGANICS	CHLORINATED DIOXIN/FURAN
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J+	The result is an estimated quantity, but the result may be biased high.	The result is an estimated quantity, but the result may be biased high.	
J-	The result is an estimated quantity, but the result may be biased low.	The result is an estimated quantity, but the result may be biased low.	
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.	The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.	The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
N		The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".	
NJ		The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	
C		This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).	
X		This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.	



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DATA ASSESSMENT

ANALYSIS: MERCURY

The current SOP HW-3c (Rev 1) September 2016, USEPA Region II for the evaluation of Mercury generated through Statement of Work ISOM02.2, and any future editorial revisions of ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time, pH (aqueous samples), or cooler temperature are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (28 days) and pH (≤ 2) have not been met, will be qualified as estimated, "J"; the non-detects (sample quantitation limits) will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery



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acceptable limits for ICV/CCV are 85 – 115%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

4. SPIKE SAMPLE ANALYSIS

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is $\geq 4x$ the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The following sample is associated with Matrix Spike sample that has spike analyte %R greater than 125%. Detects are qualified J+. Non-detects are not qualified.

Mercury MBDX61

5. DUPLICATE SAMPLE ANALYSIS

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

6. FIELD DUPLICATE

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values \geq five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is $< 5x$ the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.

Not applicable.

7. PERCENT SOLIDS



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The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. Qualifications were applied to the samples and analytes as shown below.

Not applicable.